EFFECT OF NANO SILICA ON PORTLAND SAUDI CEMENT TYPE 'G' IN HIGH PRESSURE HIGH TEMPERATURE APPLICATIONS

BY

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Dedicated to my beloved Father

Dr. Abdulkarem Ahmed Amer

And

All family members

Whose Prayers and Perseverance led to this accomplishment

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All praises and glory is to **Allah**, our God, the most beneficent, the most compassionate, and the most merciful who has given me the strength and patience to accomplish this degree.

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Thesis Abstract

Name: Sami Abdulkarem Ahmed Amer

Title: Effect Of Nano Silica On Portland Saudi Cement Type 'G' In High Pressure High Temperature Applications

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Nano silica has been introduced in recent years as an additive for concrete mixture in construction applications. Addition of Nano silica results in improved cement mechanical properties and time dependent properties. Only limited research on the application of Nano silica in oil and gas well cementing has been reported. In this study, the effect of addition of Nano silica on Portland Saudi cement type 'G' at high pressure and high temperature has been investigated. Nano silica was added at 1.0%, 2.0% and 3.0% to the cement slurry mixture currently used for oil/gas well cementing in Saudi Arabia. Experimental investigations addressed different cement slurry properties such as thickening time, fluid loss, free water separation, rheological properties, compressive strength, static gel strength, density, particles settling, and shrinkage/expansion. The results of the research showed that addition of only 1.0% and 2.0% Nano silica was feasible. Moreover, the results showed that the addition of Nano silica affected most of the addressed cement slurry properties.

خلاصة الرسالة

الإسم: سامي عبدالكريم أحمد عامر

عنوان الرسالة: تأثير مادة النانو سيليكا على الإسمنت السعودي تحت الضغط والحرارة المرتفعتين

الدرجة العلمية: ماجستير العلوم الهندسية

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تم تقديم مادة النانو سيليكا مؤخرآ كمادة مضافة الى الخرسانة في أعمال البناء. إضافة هذة المادة حسّن الخواص الميكانيكية وخواص أخرى عديدة للإسمنت. تم عمل أبحاث محدودة في مجال تطبيق مادة النانو سيليكا في سمنتة آبار الزيت والغاز. تهدف هذه الرسالة الى دراسة تأثير مادة النانو سيليكا على الإسمنت السعودي تحت ضغط وحرارة مرتفعتين. تمت إضافة مادة النانو سيليكا بنسبة 1.0%, 2.0%, و 3.0% لخليط الإسمنت المستخدم في آبار الزيت والغاز السعودية. إختبارات المعمل إستهدفت خواص الإسمنت المتعددة مثل سماكة الإسمنت, فقدان الإسمنت للسوائل, إنفصال الماء عن الإسمنت, الخواص الريولوجية للإسمنت, قوة ضغط الإسمنت, قوة الهلامية الثابتة للإسمنت, كثافة الإسمنت, ترسب جسيمات الإسمنت, و أخيرآ إنكماش / تمدد الإسمنت. نتائج البحث أظهرت أن إضافةمادة النانو سيليكا كانت ناجحة بنسب 1.0%, 2.0% فقط. أيضا, أظهرت النتائج أن إضافة مادة النانو سيليكا أثرت على معظم خصائص الإسمنت المدروسة في هذه الرسالة.

Chapter 1

Introduction

1.1 Overview

Oil and gas are considered the most important sources of energy worldwide. Obtaining this energy requires drilling thousands of wells yearly for exploration and production. Once oil or gas is found, a production strategy is planned to extract this source of energy efficiently. This involves drilling the wells as well as completing them for production and maintaining them in the future. Normally, drilling operations require running steal casing/ liner into the well to support the well bore from any collapse that could occur during drilling and also to minimize underground formation damage from drilling fluids, however, these tools must be cemented to hold in place and provide integrity to the well bore.

Cement is used in drilling operation to serve several purposes such as casing protection and support, prevention of movement of fluids behind casing, prevention of fluid entry from high permeability formations, abandoning unneeded wells and other purposes. Cement slurry is placed in the well by mixing cement powder with water at the surface before pumping it hydraulically to the desired location. [1]

During any cementing job, cement slurry properties and characteristics should meet the job requirements in terms of time and cost [2-6]. Furthermore, the cement slurry must remain pumpable long enough to allow placement and also must have enough density to overbalance the underground formation pressure. The cement slurry should also be environmental friendly and should not cause damage or contamination to underground formations. [7-13]

Oil and gas wells vary in terms of depths, downhole pressure (up to 30,000 Psi for very deep gas wells) and temperature (up to 500 °F for deep wells). Because of this wide range of depths, pressure, and temperature, cement mixture should be designed carefully. [14]

Cementing operations involve preparation of an optimum design of the cement slurry, which includes proper amount of cement powder (sacks) with the proper amount of water, which is used to provide the fluidity of the mixture. In addition, other additives are normally used to provide desired characteristics to the slurry mixture.

Nowadays, numeroustypes of additives are used in oil/gas well cementing industry including accelerating additives, lightweight additives, heavy weight additives, retarders, lost circulation control agents, filtration control agents, friction reducers, expanding additives, silica and others. [15-17]

1.2 Need for Research

The petroleum industry encounters several challenges in different areas and need more researches to yield in improvements and developments. One of the most difficult challenges associated with drilling and completion operations is assuring good cementing job. Poor cementing jobs could result in serious consequences that may jeopardize the success of any oil and gas well.[18]

Communications between zones, gas migration, undesired fluids entry to the well bore, and casing corrosion are examples of the serious consequences resulting from poor cementing jobs. Companies and the academia are continuously conducting research projects to improve and develop new cements and chemical additives that enhance cementing oil and gas wells in various environments.[19-23]

This study investigates the effect of Nano silica material on the different properties of cement slurry.

1.3 Problem Statement

Silica products (Figure 1.1 and 1.2) have been introduced long time ago and have been used in several fields that require cementing operations [24]. Their ability to improve cement properties and to sustain cement strength in high temperature application has made them a favorable mixture with cement. [25-26]

With the advancement of Nano technology, Nano Silica(Figure 1.3) has been introduced in recent years as an additive for concrete mixture in construction applications. Nano silica has improved the cement mechanical properties and showed a potential to be incorporated with the ordinary micro silica. [27-29]

Concrete is prepared and used at surface normal pressure and temperature, where properties such as compressive/tensile strengths are crucial. However, in oil/gas wells applications, cement (slurry) is subjected to a harsh environment where the temperature and pressure are much higher than those in normal concrete applications. Since the cement slurry is pumped into the wellbore; issues such as thickening time, rheology, water loss, development of slurry strength with time, cement shrinkage and formation damage are more critical than high compressive strength developed after set. How the Nano silica will affect these properties is the main focus of this study.

Figure 1.1: Silica flour

Figure 1.2: Silica Sand

Figure 1.3: Nano Silica

1.4 Thesis objectives

The objective of this thesis is to determine the effects of Nano silica on Portland Saudi cement type 'G' used in oil/gas wells at high pressure and temperature and also to determine the optimum cement mixture design with Nano silica. Specifically the following cement properties will be studied

a) Thickening time.
b) Fluid loss.
c) Free water separation.
d) Rheological properties.
e) Compressive strength.
    - Compressive strength by "crushing"
    - Compressive strength by "sonic waves"
f) Static gel strength.
g) Density.
h) Particles settling.
i) Shrinkage/Expansion.

After studying the above cement key properties, the following will be achieved

1. Determination of the effects of Nano silica on cement slurry at high pressure high temperature environment.
2. Identification of the applicable percentage range of Nano silica that can be used for cement mixture design.

3. Identification of the applicability of mixing the Nano silica with other silica products such as silica flour and silica sand.
4. Identification of the impact of Nano silica on the behavior of other cement additives such as retarder, dispersants… etc.

1.5 Research approach

In this study, a typical well in Saudi Arabia is selected and the following will be conducted:

1. Current cement slurry design will be studied and subjected to the following testing process
    - Thickening time test
    - Fluid loss test
    - Free water separation test
    - Rheological properties measurements
    - Ultrasonic cement analysis
    - Pressurized density measurements
    - Compressive strength test "crushing"
    - Static gel strength analysis
    - Particles settling test
    - Expansion/Shrinkage test
2. Different percentages of Nano Silica material will be added to the cement mixture and the testing process will be repeated.

3. Comparison between the cement systems behavior with and without the Nano silica will be made, and cement system that exhibits the best behavior will be determined.
4. Conclusions will be drawn from this study and recommendations for future work will be suggested.

1.6 Thesis organization

The organization of this thesis is as follows

Chapter 2: presents an overview of oil and gas well cement system and its components. Moreover, this chapter gives the reader detailed information about cement properties and additives and also discusses the factors that influence cement slurry design.

Chapter 3: presents a literature survey of the researches that have been previously conducted on cement slurry development and chemical additives improvements over the years.

Chapter 4: presents a detailed description of the experimental program planned for this research. The experimental program consists of several cement tests that each is aimed to address certain cement property to be studied.

Chapter 5: presents the results of all cement tests of the experimental program with detailed analysis and discussion.

Chapter 6: concludes the thesis work and highlights research major outcomes with recommendations for future work.

Chapter 2

Cement system

2.1 Cement system components

Cement system is defined as the cement slurry design consisting of different materials that each is used to give certain property for performance improvement. The cement system normally consists of the cement powder (Portland cement) which is considered as the main element and is made of chemical compounds such as calcium silicate as well as calcium aluminate with other oxide components.

The second cement system element is water that is mixed with the cement powder to provide the fluidity to the mixture and also to act as a hydration agent to the cement. Water must be optimized when added to cement and the water to cement ratio must be carefully selected since low water to cement ratio results in high cement slurry viscosity and rate of set, while high water to cement ratio may cause free water separation and a reduction in cement density.

The third cement system element is the chemical additives which assist cement system performance by adding desired properties to the cement.

2.2 Cement classifications

There are variety of cement types and classes used worldwide in different fields and applications. However, the petroleum industry has accepted the eight cement classes set by the American petroleum institute (API). The cement classes are designated by letters from (A to H) intended for certain job specifications. The following table explains the different API cement classes with its corresponding applications.

Cement class	**Cement application**
Class A	Intended for use from surface to a depth of 6,000 ft when special properties are not required
Class B	Intended for use from surface to a depth of 6,000 ft when conditions require moderate to high sulfate resistance
Class C	Intended for use from surface to a depth of 6,000 ft when conditions require high early strength. Available in ordinary type and in moderate and high sulfate resistant types.
Class D	Intended for use at depths from 6,000 to 10,000 ft and at moderately high temperatures and pressures. Available in both moderate and high sulfate resistant types.
Class E	Intended for use at depths from 10,000 to 14,000 ft and at high temperatures and pressures. Available in both moderate and high sulfate resistant types.
Class F	Intended for use at depths from 10,000 to 16,000 ft and at extremely high temperatures and pressures. Available in high sulfate resistant types.
Class G	Intended for use as basic cement from the surface to a depth of 8,000 ft as manufactured. With accelerators and retarders it can be used at a wide range of depths and temperatures. It is specified that no additions except

	calcium sulfate or water, or both, shall be interground or blended with the clinker during the manufacture of Class 'G' cement. Available in Moderate and High Sulfate Resistant types.
Class H	Intended for use as basic cement from the surface to a depth of 8,000 ft as manufactured. This cement can be used with accelerators and retarders at a wide range of depths and temperatures. It is specified that no additions except calcium sulfate or water, or both, shall be interground or blended with the clinker during the manufacture of class H cement. Available only in moderate sulfate resistant type.

Table 1: API cement classes [1]

It should be noted that the cement used in this study is Portland Saudi cement type 'G' that complies with API classifications standards.

2.3 Cement additives

The performance of the cement system can be controlled by chemical additives, thus, the cementing job can be conducted as desired. There is a wide range of chemical additives produced by different companies to meet petroleum industry requirements. The following subsections review the most common used cement additives that influence cement slurry design.

2.3.1 Density control agents

The first type of cement additives considered here is the density control agent. This type of additives is used to increase or decrease cement slurry density. Increasing cement slurry is needed to overbalance formation pressure preventing fluid flow from the formation to the well bore. Noting that excessive increase in cement slurry density may increase the hydrostatic pressure higher than the formation pressure resulting in fluid loss into the formation and sometimes undesired fractures.

The most common density control additives are barite and hematite which are considered as weighting agents to increase cement slurry density. On the other hand, the most common lightening weight agents are pozzolans and nitrogen.

2.3.2 Acceleration/Retardation agents

Accelerators and retarders are used to control cement slurry thickening time during cementing operation. This is extremely important to make the cement slurry pumpable until it reaches its destination in the annular space between formation and the steel casing.

Accelerators are used for shallow low temperature and pressure cement jobs where long thickening time is not necessary while retarders are used for deep high temperature and pressure cement jobs where thickening time needs to be adequate to complete the cementing operation safely.

The most common accelerators used are calcium chloride and sodium chloride while the most common retarders used are calcium lignosulfonates and borax.

2.3.3 Fluid loss control agents

Controlling fluid loss rate is an important issue to be considered when cementing across permeable formations where it could be damaged by the cement slurry filtrate.

Therefore, fluid loss control agents are used to control the rate at which the slurry loses water and maintain it within the acceptable industry standards.

The most common fluid loss agents used are the organic polymers and cellulose derivatives.

2.3.4 Lost circulation control agents

Controlling lost circulation is an important issue to be considered when cementing across highlypermeable and vuggy formations as well as formations having natural or induced fractures.

Lost circulation might be controlled by reducing cement slurry density and by adding additives to act as a plugging bridge on the opening area of the high permeability zone or the fracture.

There are different types of lost circulation control agents, granular type(e.g. Gilsonite), flake type (e.g. cellophane), and fibrous agents (e.g. nylon).

2.3.5 Defoaming agents

Cement foaming is one of the problems associated with cement slurry while mixing. The entrapped air in the cement slurry could cause damage to the pumps in the field and also could cause incorrect density readings and consequently mixing incorrect cement slurry density.

Defoamers are used to minimize foaming problems and are normally used with every cement system. Defoamers are special additives developed by different companies and are available in powder or liquid for convenient use.

2.3.6 Dispersion agents

Dispersants are used mainly to control cement slurry rheological properties for better mixing and pumping. They have the ability to reduce friction between cement slurry particles resulting in lower pumping pressure requirement and also reducing water to cement ratio which improves cement compressive strength. In addition, dispersants are used to create turbulent flow for improved mud removal from the wellbore. However, it has been notice that dispersants have the tendency to make cement slurry thickening time longer, so it should be used with caution.

Dispersants are special additives developed by different companies and are available in powder or liquid for convenient use.

2.3.7 Expansion agents

Cement slurry is pumped downhole and placed in the annular space between the casing/liner and formation. After cement set, the cement column may suffer from shrinkage due to the harsh environment downhole in terms of pressure and temperature. Therefore, expansions additives are used to give cement an expansion property to bond with the casing/liner and to give integrity to the well bore.

Moreover, cement expansion can provide better zonal isolation, minimize gas migration and fluid entry problems, and prolong the productivity of wells.

2.3.8 High temperature agents

Researches and field practices have shown that cement under high temperature (> 230 °F) tends to suffer from compressive strength retrogression which consequently jeopardizes the productivity of the well. Therefore, silica products such as silica flour and silica sand are added to the cement system to sustain compressive strength at high temperature over long period of time.

Chapter 3

Literature Review

Extensive work has been done on cement system and its associated problems. However, only few researches have been conducted on the area of Nano particles incorporation with cement and the main focus was from other fields such as civil and chemical engineering.

Different areas are included in this survey covering development of cement systems through the development of cement additives, cement testing techniques, field studies of cement systems, and the evolution of Nano particles additives to cement systems.

A.S.Dahab and A.E.Omar (1989) [30]: investigated the suitability of Saudi cement in oil well cementing. They conducted chemical and physical analysis on cement powder, rheology and thickening time tests on cement slurry, andcompressive strength testing on cement set. They concluded that the rheological properties of cement slurries exhibited pseudoplastic behavior regardless of the type of water and cement additives. Thickening time and compressive strength depend on the type and percentage of additives used and the set cement stability was overall good since the set cement reached 500 psi pressure in 24 hours. (Figure 3.1)

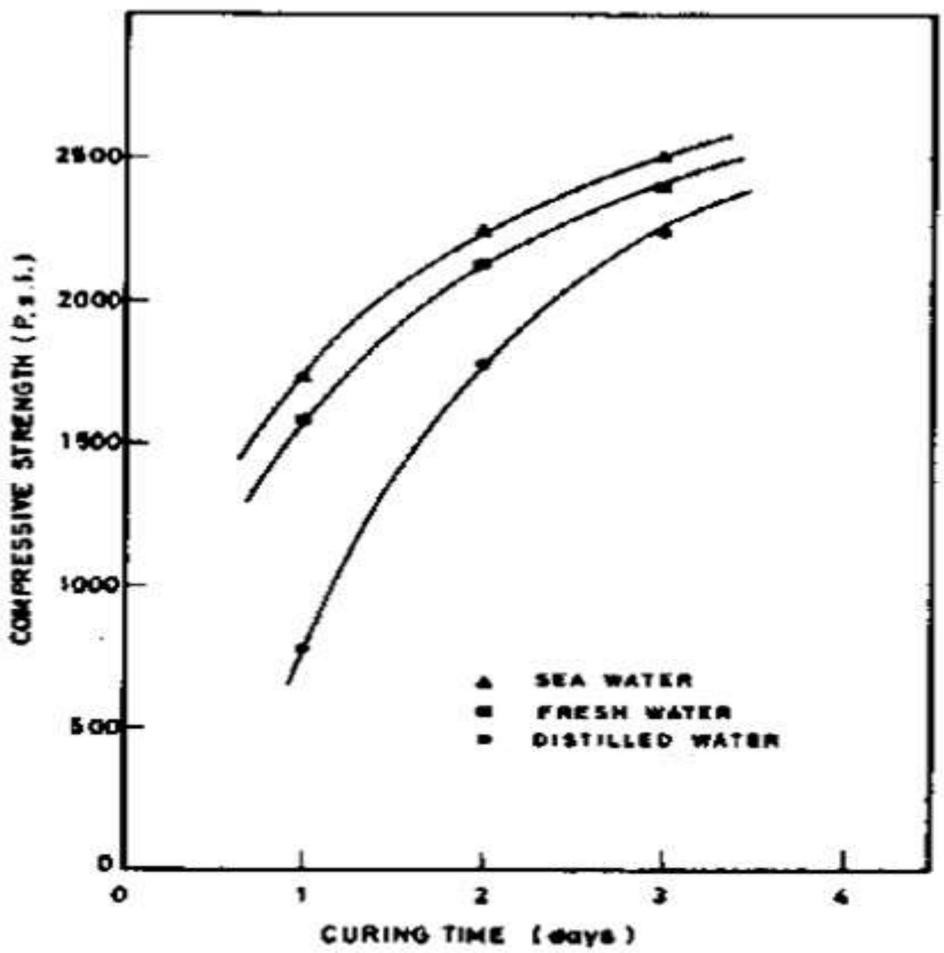


Figure 3.1: Compressive strengths of Saudi cement systems having different types of water [30]

Jones & Carpenter (1991) [4]: presented a combined (latex and thixotropic) cementing system that improves cement bonding and zonal isolation in wells havingbottomhole static temperature of 175 °F. Laboratory studies and case histories indicated that adopting this system will yield in better primary and remedial cementing through gas migration prevention, high degree of zonal isolation, filtrate loss reduction with rapid compressive strength gain, and minimal waiting on cement.

E. B. Nelson and J. M. Casabonne (1992) [31]: presented a slurry design method to address the problem of achieving adequate slurry placement time and also short wait on cement time for deep wells. He mentioned in his modified slurry design procedure that compressive strength of ≥ 500 psi must be reached within 24 hours at static temperature at top of cement and also safety margin must be considered to prevent premature set in case of any operation shutdown.

K.R.Backe et al. (1997) [32]: studied the transition period of curing oil well cements where they showed that the cement curing characteristics are function of temperature and there is a correlation between shrinkage and cement content. Mechanism of gas migration was proposed in their work where the gas has to overcome the entry pressure of the cement pores and fracture the cement structure. They also indicated that gas migration can be predicted based on gas tightness factor defined in their paper.

K.R.Backe et al. (1998 and 1999) [33, 34]: investigated the characteristics of curing cement slurries by electric conductivity which they claimed that it can be considered as a parameter for monitoring the entire hardening process of oil well cement. They related the electrical conductivity of oil well cement slurries to cement chemistry and physical properties. Also, they established an indirect relationship between conductivity and compressive strength obtained from ultrasonic cement analyzer and also presented a conservative drillout strength criterion based on conductivity. Furthermore, they claimed that there is relationship between electrical conductivity and porosity by the use of Archie's law. In their other paper under the same title, they observed that adding silica fume will reduce the transition period significantly.

Jeff Hibbeler et al. (2000) [35]: reported a laboratory data to support the use of local cements as an alternative to imported API class G cement. Case studies were also presented of the use of local cement in oil well applications as a way to save drilling costs and help local economies. Moreover, they indicated that consistency and performance are the critical parameters that govern the usefulness of particular cement.

Thomas Heinold et al. (2002) [36]: studied the effect of key cement additives on the mechanical properties of normal density cement. Organic and non-organic material were added to oil field cements with water to cement ratio of (0.5-0.66) and then the cements were subjected to unconfined compressive strength tests as well as tensile and flexural strength tests to examine the effect of those additives. They concluded that using the additives individually will not enhance the flexural and tensile strength properties in high density cement. Moreover, additives known to improve flexural and tensile strengths properties in normal density may not work effectively in higher density and temperature cement systems.

HuiLi et al. (2004) [28]: conducted an experimental study to investigate the compressive/ flexural strengths of Nano iron oxide (Fe_2O_3)and Nano silica (SiO_2) cement mortars. They noticed that cement mortars having these Nano materials showed higher compressive/flexural strengths when compared to the plain cement mortars.

In the same area, **Byung Wan Jo et al. (2005) [37] and Jeng. Y. S et al. (2006) [38]** conducted similar experiments to cement mortars having Nano silica and they observed that Nano silica is more effective in enhancing cement compressive strength than the ordinary silica fume. (Figure 3.2)

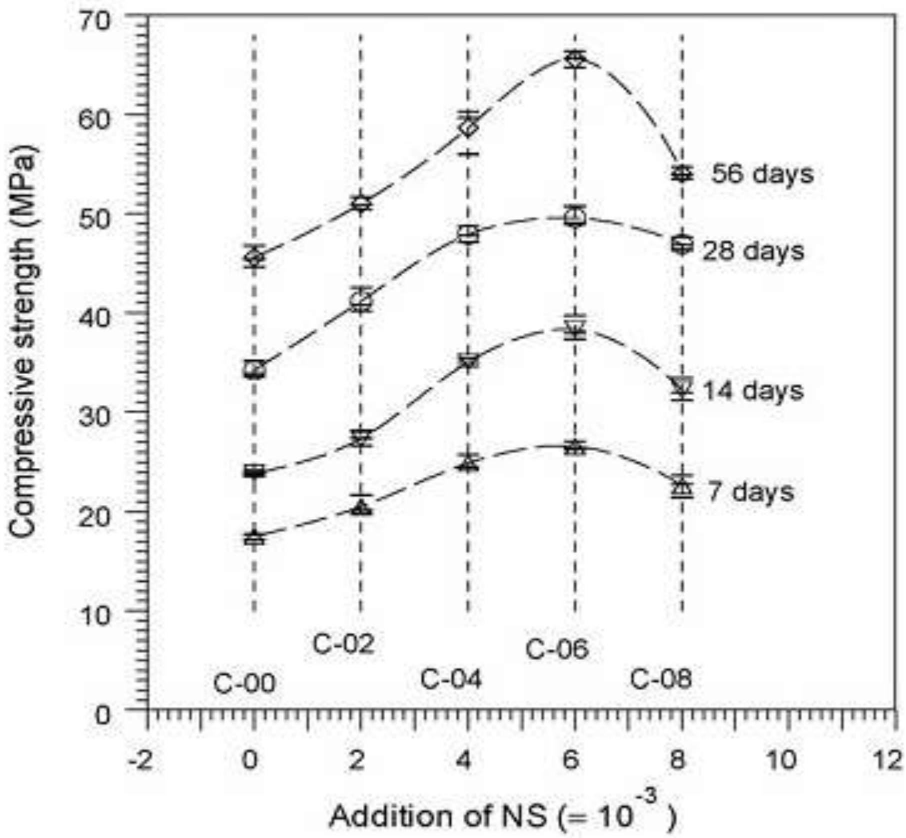


Figure 3.2: Compressive strengths of Portland cement composite at various percentages of Nanosilica [38]

S. Jennings (2005) [39]: conducted long term high temperature cement test to investigate the durability of Saudi cement. In his study, he performed several tests to address cement properties such as compressive strength, permeability, shrinkage/ expansion, rheology, settling, Poisson ratio, and Young's modules. He concluded that the cement shrinkage was high (Figure 3.3), compressive strength of cement declined by 81 % in 11.75 months when cured at 300 °F and 3000 psi (Figure 3.4). Moreover, he noticed the permeability has increased 7.7 fold in one year and Young's modules decreased 81 % in one year. (Figures 3.5 and 3.6)

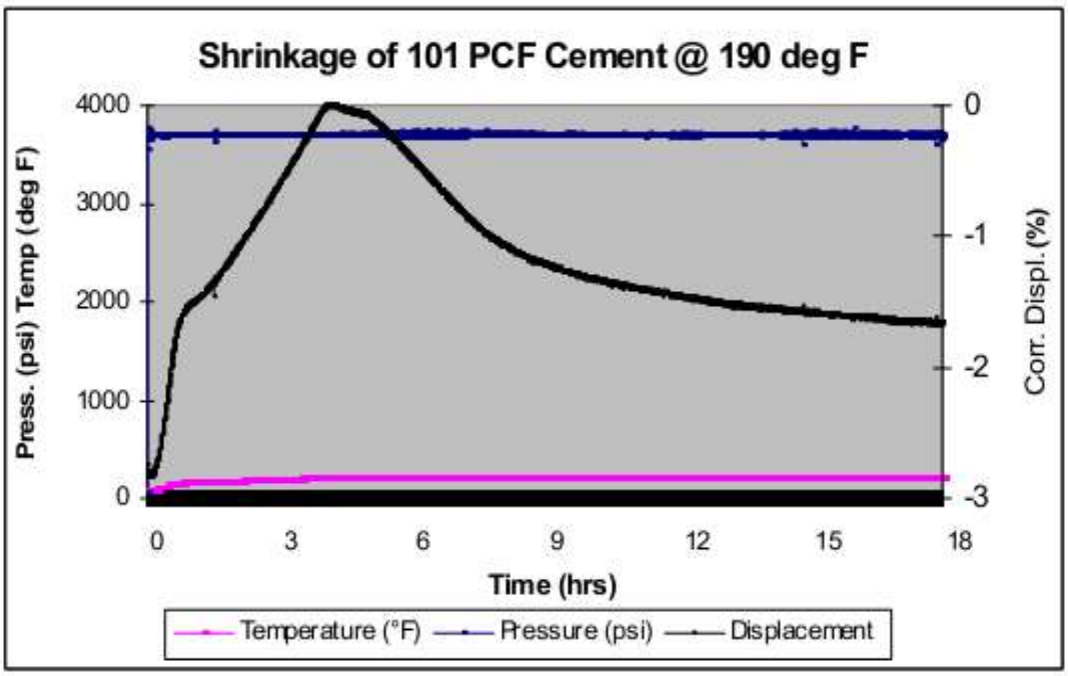


Figure 3.3: Shrinkage of Saudi cement at 190 °F[39]

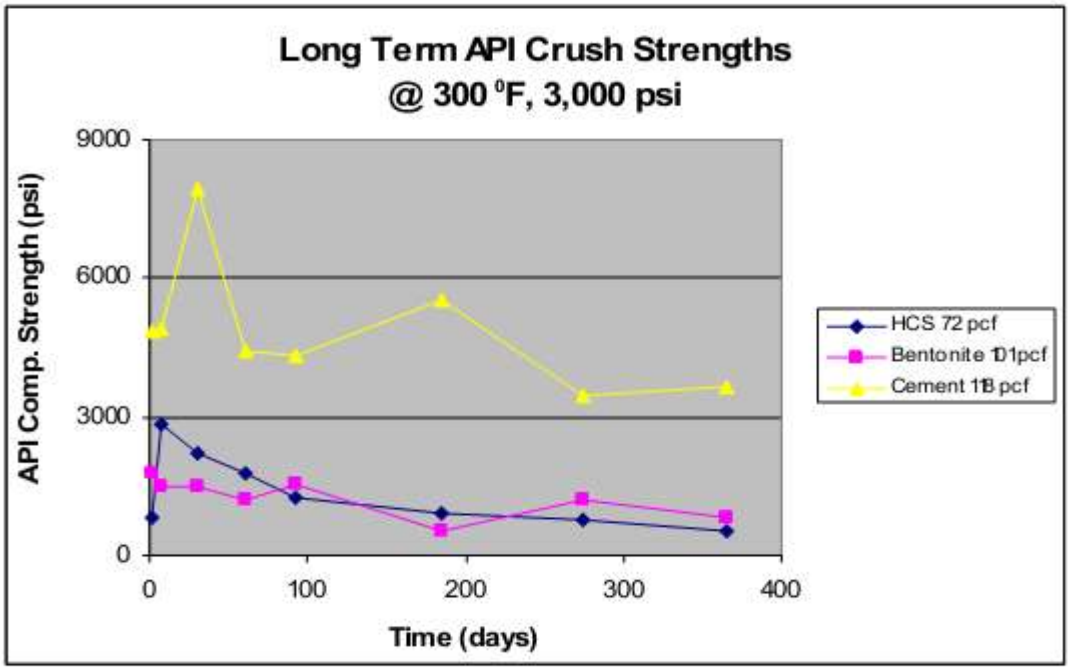


Figure 3.4: Compressive strength of Saudi cement at 300 °F and 3000 Psi[39]

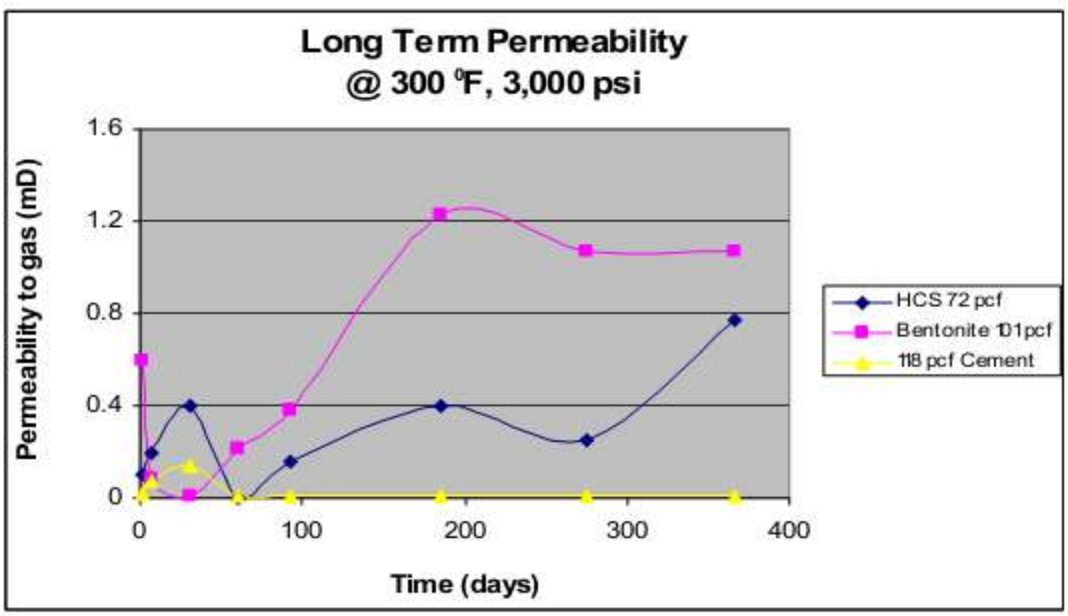


Figure 3.5: Permeability of Saudi cement at 300 °F and 3000 Psi[39]

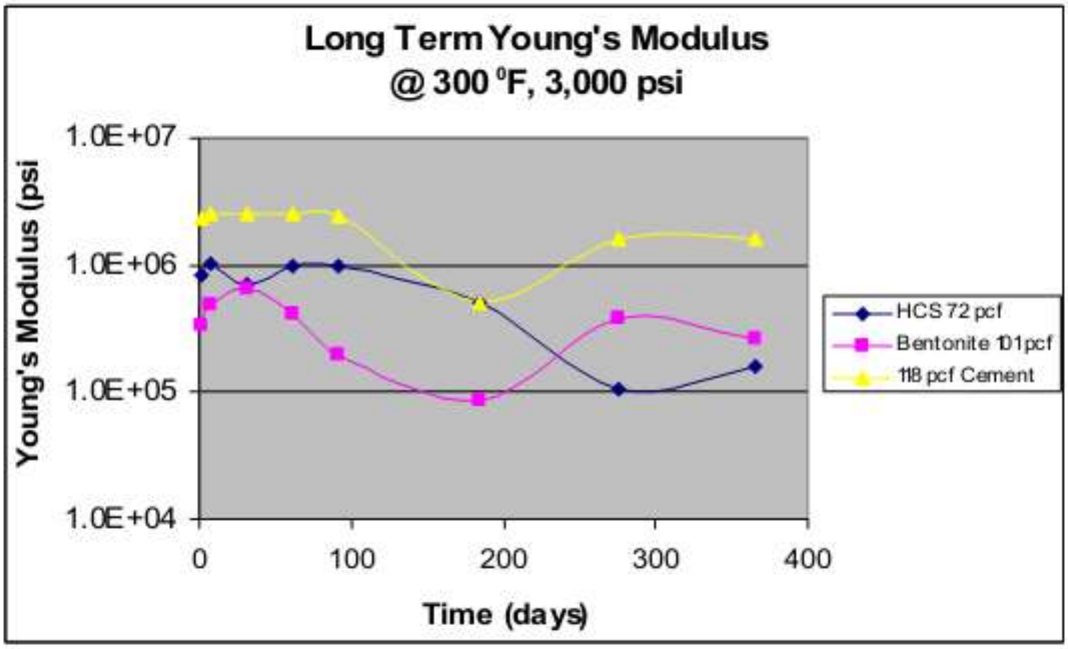


Figure 3.6: Young's Modulus of Saudi cement at 300 °F and 3000 Psi[39]

Al-Yami et al. (2006, 2008) [21, 23]: introduced a cement system that yields in a slow rate of penetration for the use in drilling side-track wellbores. They observed that increasing cement density reduces the rate of penetration slightly for side tracking drills and also as compressive strength increases, the rate of penetration decreases.

Y. Qing et al. (2007) [40]: studied the effect of Nano silica on the properties of cement pastes where they conducted several tests such as compressive strength and setting time tests. They indicated that the influence of Nano silica and silica fume is different since Nano silica makes cement pastes thicker and also accelerates the hydration process. In terms of compressive strength, they noticed that compressive strength increases with increasing Nano silica percentage.

B.R.Reddy et al. (2009) [41]: conducted a comparative study of laboratory methods to measure cement shrinkage in environment where cement slurry is exposed to open or closed external water source. They used recommended API test apparatus such as balloon and ring molds under atmospheric condition and flask method under pressure condition. Conclusion drawn out of their work that the flask, balloon, and ring molds methods are good enough to study the relative inherent tendencies of slurry compositions to shrink under ambient condition. However, they don't represent bulk shrinkage similar to that occurring in the wellbore even though the volumetric changes obtained from the ring molds represent the restraining effect on cement composition but may not be suitable of measuring the chemical shrinkage by curing under water.

L. Senff et al. (2009) [27]: investigated the effect of Nano silica on rheology and fresh properties of cement pastes and mortars. They conducted rheological tests and showed that after (75) minutes from mixing start, mortar having (2.5%) "By weight" Nano silica suffered from insufficient flowability. They also found that Nano silica increased the yield stress by (66.5%) and plastic viscosity by (3.6%). They also found that setting time and moment to reach maximum temperature decreased by (60%) and (51.3%) respectively when compared to the mixture without Nano silica added. Finally, they reported that calcium hydroxide was formed after 9 hours in the sample having (2.5%) "By weight" Nano silica. Also the air content increased by (79%) and apparent density decreased by (2.4%) after adding Nano silica.

L. Sneff et al. (2010) [42]: continued the investigation on the effect of Nano silica and silica fume on rheology, compressive strength, shrinkage, weight loss and other properties of mortars. In their study, Nano silica percentage was (0-7) % by weight and silica fume percentage (0- 20) % by weight with water to binder ratio of (0.35 – 0.59). They concluded that mortar having (7%) Nano silica exhibited faster structures formation during Rheology test which influenced the yield stress. Moreover, they reported that apparent porosity reached maximum for mortars with (7%) Nano silica and the increase of shrinkage percentage over the silica fume was (80%) after7 days and (54%) after 28 days.

Dossary 2011 et al. (2011) [18]: investigated the causes of a poor cementing job in one onshore field in Saudi Arabia. In their study, they reviewed field practices reports, well logs, and conducted several experiments to simulate the same downhole conditions over the same period of time when the communication problem between two formations occurred. Eventually, they discovered that the root cause of the communication problem was the loss of hydrostatic pressure before cement developed enough compressive strength after cement placement.

V. Ershadi et al. (2011) [43]: investigated the effect of Nano Silica on Iranian class G cement. Water to cement ratio used in their study was 0.6 and slurry density of 100 pound per cubic feet (Pcf) and they focused on the effect of Nano silica on porosity, permeability, and compressive strength. They noticed that by adding Nano silica, porosity and permeability decreased by 33.3 % and 99 % respectively (Figure 3.9).

Compressive strength was tested using the ultrasonic cement analyzer at T=158 °F and P=3000 Psi and growth of compressive strength (Figure 3.8) was noticed after adding the Nano Silica. Effect of Nano Silica on cement Rhoeolgy was also reported where increasing the amount of Nano silica will decrease slurry density; reduce free water and fluid loss (Figure 3.10), increase plastic viscosity (Figure 3.11) and yield point, and decrease thickening time (Figure 3.7).

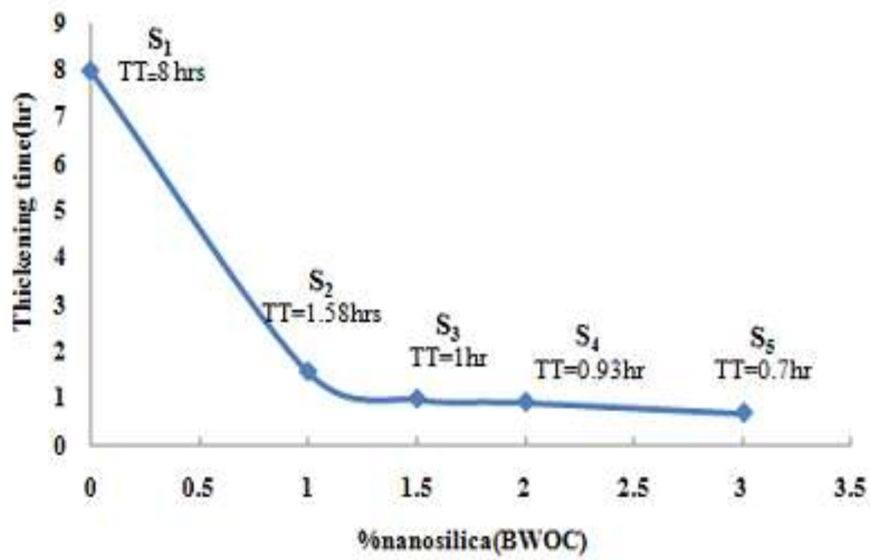


Figure 3.7: Reduction in thickening time by adding Nano silica [43]

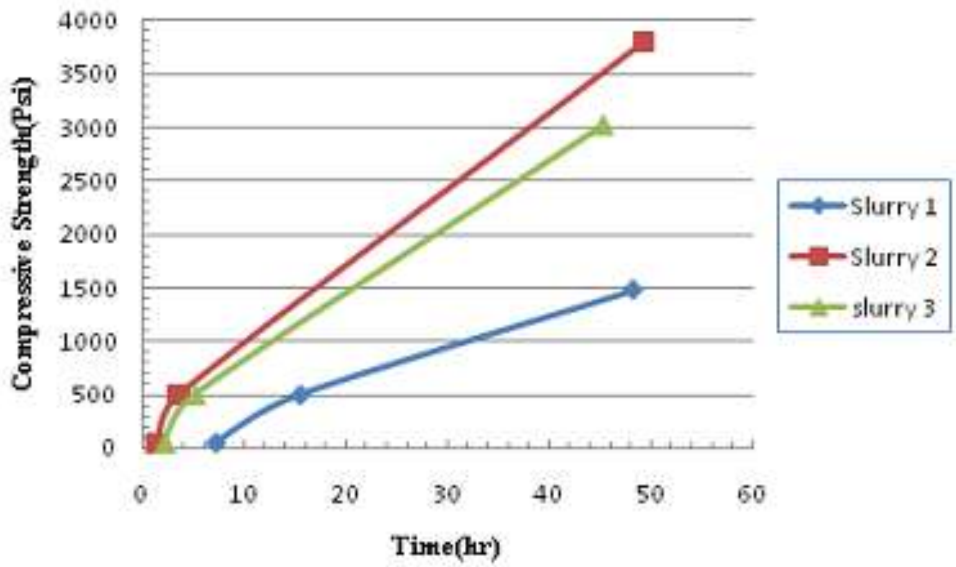


Figure 3.8: Changes in compressive strength with time [43]

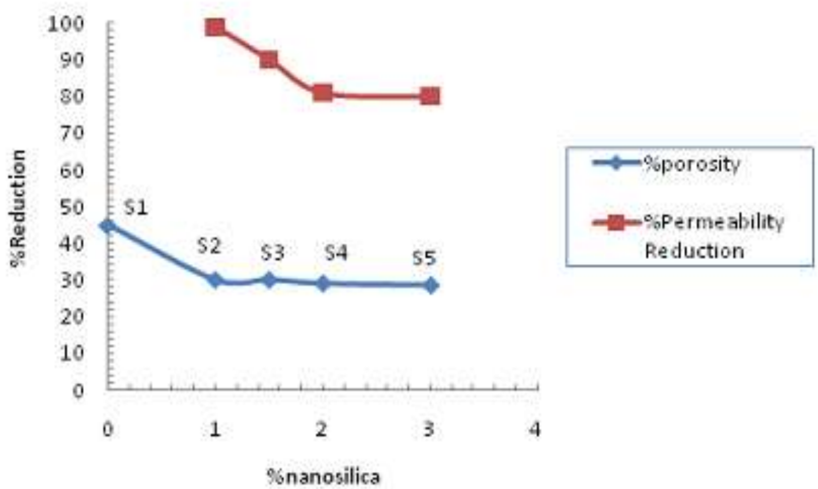


Figure 3.9: Percentage of porosity and permeability reduction [43]

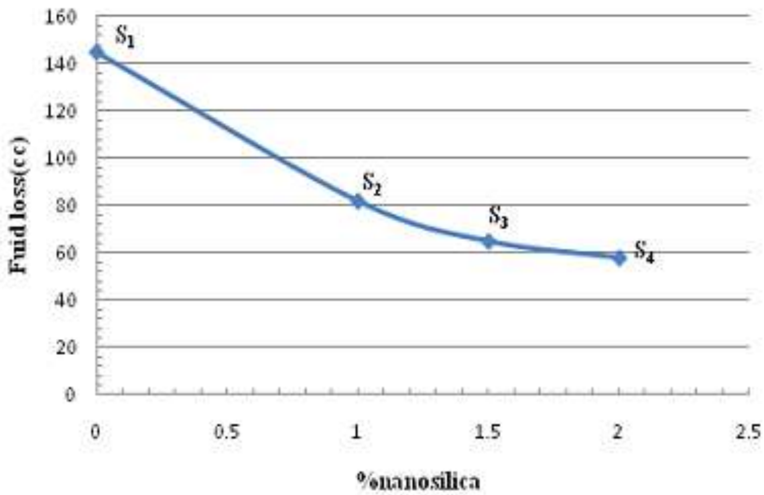


Figure 3.10: Reduction in fluid loss by adding Nano silica [43]

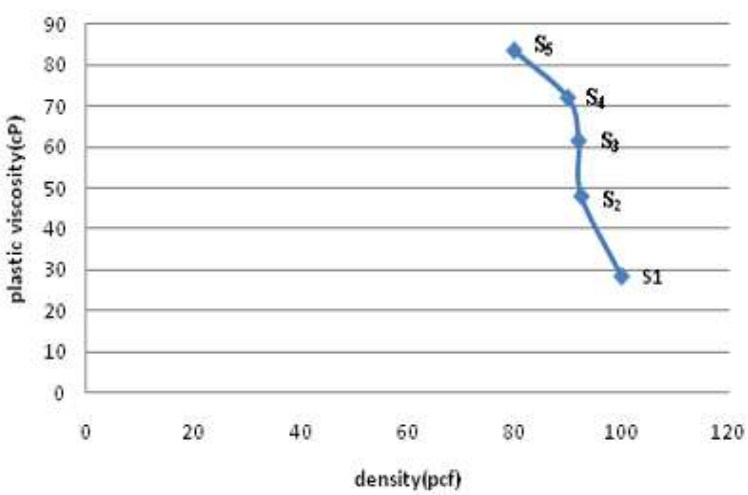


Figure 3.11: Change in plastic viscosity with density [43]

Similarly, **M. Hong Zhang and Jahidul Islam (2012) [44]** and **M. Chooleui et al. (2012) [45]:** studied the effect of Nano silica on cement mortars and reached to the same conclusions (Rheology improvement, increase in compressive strength, No free water, decrease in setting time, porosity and permeability) as obtained earlier by previous authors.

Chapter 4

Experimental Program

Cement lab testing is an important process to evaluate and develop different properties of cement system and to attempt to mimic the actual behavior of the cement in high pressure high temperature downhole environment.

The experimental program planned for this study is implemented according to the American petroleum institute (API) procedures[46] and consists of several cement tests that each is aimed to address certain cement property. The cement properties included in this program are:

a) Thickening time.

b) Fluid loss.

c) Free water separation.

d) Rheological properties.

e) Compressive strength.

   - Compressive strength by "crushing"
   - Compressive strength by " sonic waves"

f) Static gel strength.

g) Density.

h) Particles settling.

i) Shrinkage/Expansion.

Thus, the effect of Nano silica on these cement properties will be investigated.

4.1 Well Specifications

A typical well in Saudi Arabia is selected having the following specifications:

Job Type: cementing 7" inch liner

Depth: 14,000 feet

Bottom hole circulating temperature (BHCT): 228 °F

Bottom hole static temperature (BHST): 290 °F

Time to reach bottom (TRB): 49 minutes

Surface pump pressure: 1050 Psi

Mud Weight: 85 PCF

Bottom hole pressure: 8265 Psi

4.2 Cement system consideration

The selected well has a special cement system since the well is deep with high pressure and temperature conditions. The cement system consists of different materials each contributes and adds chemical and physical property to make the cementing job successful.

The following table contains the materials of the cement design (1**) without** the addition of the Nano silica material.

Class G cement powder + 35% silica flour + 1.0 % Expanding agent + 0.4 % Dispersant + 0.2 % Fluid loss control agent 1 + 0.5 % Fluid loss control agent 2 + 1.0 % Retarder +0.25 gmDefoamer	
Slurry Density (Expected) , PCF	125
Water to cement Ratio	0.435
Slurry Yield , ft^3/Sack	1.367
Thickening Time (Expected)	4 – 5 hours

Table 2: Cement Slurry design without Nano silica

This cement slurry system will be processed through a series of cement tests in the experimental program to obtain results that will be considered as the base case to be referred to.

Next, the Nano silica is introduced to the above cement system, thus, slurry design **with** the addition of Nano silica will be as follows:

Class G cement powder + 35% silica flour + X % Nano silica + 1.0 % Expanding agent + 0.4 % Dispersant + 0.2 % Fluid loss control agent 1 + 0.5 % Fluid loss control agent 2 + 1.0 % Retarder + 0.25 gmDefoamer	
Slurry Density (Actual), PCF	Unknown
Water to cement Ratio	0.435
Slurry Yield , ft^3/Sack	Unknown
Thickening Time (Expected)	Unknown

Table 3: Cement Slurry design with Nano silica

Where X in table (3) represents Nano silica percentages to be used. The plan is to investigate the effect of Nano silica percentages of (0 %, 1.0 %, 3.0 %, and 5.0 %) by cement weight.

4.3 Thickening Time Test

- **Purpose**

To determine the time the cement slurry will reach 100 Bearden unit of consistency (BC) which is considered the length of time the slurry remains pumpable.

- **Testing Apparatus**

The pressurized consistometer, (Figure 4.1) is the most common machine used to determine the thickening time of cement slurry while being stirred under high pressure and temperature conditions.

Figure 4.1: Pressurized consistometer

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes. (Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. The slurry is poured into the consistometer slurry cell and placed inside the machine to be stirred at 150 rpm, 228 °F, and 9315 Psi.
7. Slurry consistency is monitored and time to reach consistency of 100 Bc is recorded.

Figure 4.2: Cement slurry mixing jar

Figure 4.3: Silica flour

Figure 4.4: Mixing cement powder with silica products

Figure 4.5: Cement Slurry mixing

Figure 4.6: Mixing cement powder with silica products

4.4 Fluid Loss Test

- **Purpose**

To determine the amount of fluid loss from the slurry under high pressure and temperature conditions.

- **Testing Apparatus**

High pressure High temperature cement slurry fluid loss tester.

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes. (Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. After that, the slurry is conditioned for 20 minutes at 194 °F temperature and atmospheric pressure. (Figure 4.7)
7. After conditioning, the slurry is taken to the fluid loss testing cell to be tested under 1000 Psi and 194 °F for 30 min. (Figure 4.8)
8. The amount of collected fluid loss is measured. (Figure 4.9)

Figure 4.7: Atmospheric consistometer

Figure 4.8: HP/HT fluid loss tester

Figure 4.9: Collected Slurry fluid loss

4.5 Free water separation Test

- **Purpose**

To determine the amount of free water that forms on top of the cement slurry column after certain period of aging time.

- **Testing Apparatus**

Graduate cylinder

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes.(Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. After that, the slurry is conditioned for 20 minutes at 194 °F temperature and atmospheric pressure. (Figure 4.7)
7. Next, the slurry is carefully poured into a graduate cylinder. (Figure 4.10)
8. Aluminum foil is used to cover the top of the cylinder and the slurry is aged for 2 hours.

9. Finally, the free water separated is drained using a syringe and the percentage of free water is calculated.

Figure 4.10: Graduate cylinder for free water test

4.6 Rheology Test

- **Purpose**

To determine different rheological properties such as viscosity and yield Point of cement slurry under high temperature condition.

- **Testing Apparatus**

The most common equipment used to measure the rheological properties is the variable speed Rheometer. (Figure 4.11)

Figure 4.11: Variable speed Rheometer

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes.(Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. After that, the slurry is conditioned for 20 minutes at 194 °F temperature and atmospheric pressure. (Figure 4.7)
7. Next, the slurry is poured into the Rheometer cup that was preconditioned at the same temperature 194 °F.
8. The slurry is stirred for about 10 seconds at 300 rpm, 200 rpm, 100 rpm, 6 rpm, and 3 rpm.
9. Viscosity readings are recorded at every speed.

4.7 Compressive Strength Test

- **Purpose**

To determine the cement capacity to withstand axial pushing forces.

Two methods will be employed to determine the cement compressive strength, direct method by applying physical forces to square inch cement cubes and indirect method utilizing ultrasonic waves that cement slurry will be subjected to.

4.7.1 Compressive Strength test '' Crushing ''

- **Testing Apparatus**

The most common machine used for compressive strength determination is the API compressive strength tester. (Figure 4.12)

Figure 4.12: Compressive strength tester

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes.(Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. Next, cement slurry is carefully poured into molding cubes to make square shape samples (2 inch x 2 inch).(Figure 4.13)
7. After that, the sample molds are taken to the HP/HT curing chamber to be conditioned for 24 hours at 290 °F temperature and 3000 Psi pressure. (Figure 4.14)
8. Cement samples are taken to the compressive strength test. (Figure 4.15)
9. Cement sample will be subjected to gradually increasing axial force until sample fails and the maximum load applied is recorded.

Figure 4.13: Cement molds

Figure 4.14: Cement samples being cured in the HP/HT curing chamber

Figure 4.15: Cement cubes to be tested

4.7.2 Compressive Strength test '' Ultra sonic ''

- **Testing Apparatus**

Ultrasonic Cement analyzer(UCA)

Figure 4.16: Ultrasonic Cement Analyzer (UCA)

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes.(Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. After that, the slurry is conditioned for 20 minutes at 194 °F temperature and atmospheric pressure. (Figure 4.7)
7. Finally, cement slurry is carefully poured into a special cell before inserting it in to the UCA**.** (Figure 4.17)
8. The UCA is set to bottom hole circulating temperature of 228 °F in the first 49 minutes and then temperature increases to bottom hole static temperature of 290°Fand pressure of 4666 psi.
9. UCA is run for 24 hours and the development of cement sonic strength is monitored.

Figure 4.17: Slurry cell for the UCA

4.8 Static gel strength Test

- **Purpose**

To determine static gel strength of cement slurry that gives an indication of the development of slurry gellation process with time.

- **Testing Apparatus**

Static gel strength analyzer (SGSA) (Figure 4.18)

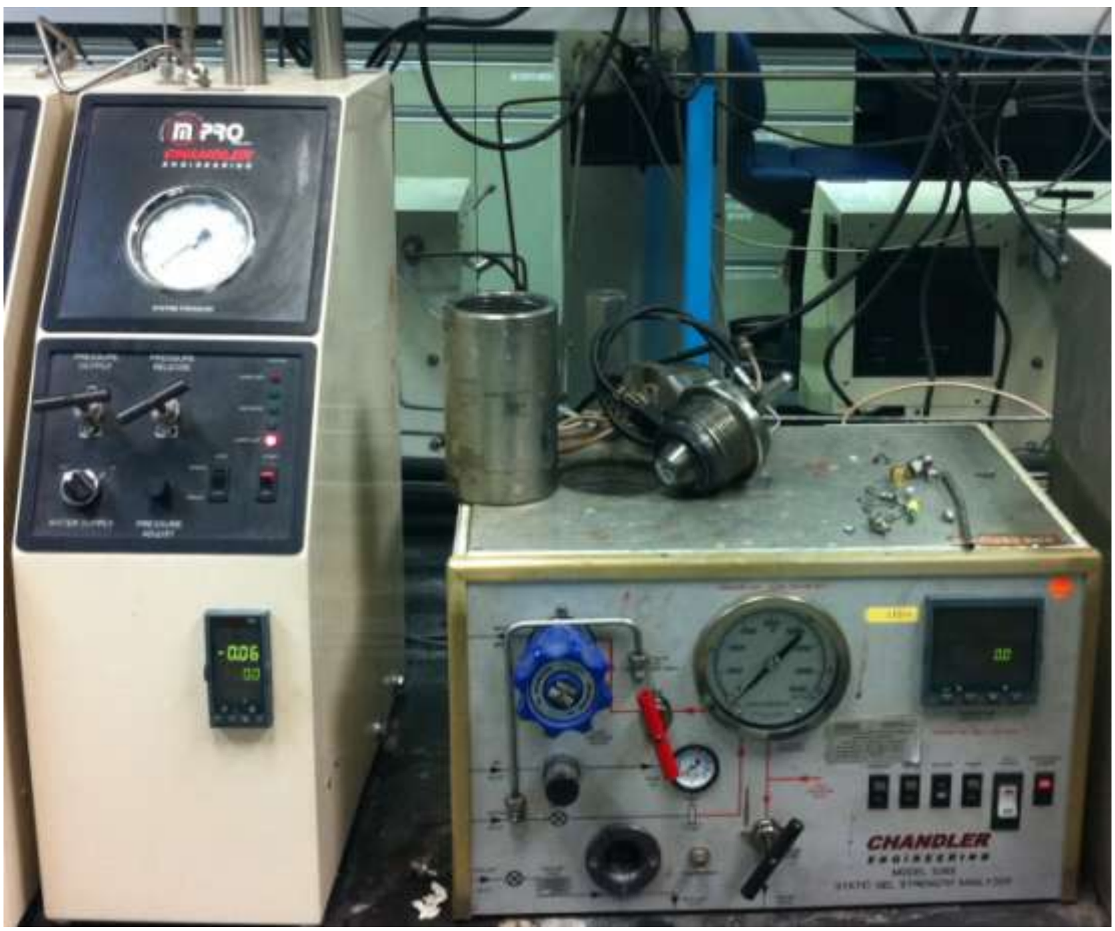


Figure 4.18: Static gel strength analyzer (SGSA)

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes. (Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. After that, the slurry is conditioned for 20 minutes at 194 °F temperature and atmospheric pressure. (Figure 4.7)
7. Finally, cement slurry is carefully poured into a special cell before inserting it in to the SGSA**.** (Figure 4.17)
8. The SGSA is set to bottom hole circulating temperature of 228 °F in the first 49 minutes and then temperature increases to bottom hole static temperature of 290°Fand pressure of 4666 psi.
9. SGSA is run for 24 hours and the development of cement compressive and gel strengths are monitored.

Note: SGSA testing procedure is similar to the UCA since SGSA gives compressive strength data as well.

4.9 Density Measurement

- **Purpose**

The purpose of this test is to determine of the actual density of cement slurry under pressure condition.

- **Testing Apparatus**

The most common used tool to measure the density of any fluid is the Pressurized density balance that is superior to the normal density balance in terms of reducing entrapped air and the correlating error. (Figure 4.19)

Figure 4.19: Pressurized density balance

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes. (Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. The densometer is calibrated in prior, and then the slurry is poured fully into the density balance cup and sealed carefully.
7. The slurry is sealed inside the cup and pressure is applied.
8. The rider is then moved until the bubble in the level glass is centered.
9. Corresponding density is read and recorded.

4.10 Particles Settling Test

- **Purpose**

To determine cement particles settling that could take place by means of density segregation.

- **Testing Apparatus**

Special long cells are used to allow cement particles settling effect to take place over certain aging period of time. (Figure 4.20)

Figure 4.20: Cement particles settling cells

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes. (Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. After that, the slurry is conditioned for 20 minutes at 194 °F temperature and atmospheric pressure. (Figure 4.7)
7. Next, cement slurry is carefully poured into the long cells that were pre-heated at the same temperature 194 °F.
8. The cells are then taken to the HP/HT curing chamber to be conditioned for 24 hours at 290 °F temperature and 3000 Psi pressure. (Figure 4.14)
9. The cement samples are extracted from the cells and cut into three pieces (top, middle, and bottom) for density segregation measurements. (Figures 4.21 and 4.22)
10. Cement pieces (top, middle, and bottom) are weighted before and after immersing in water and the difference is noted. (Figure 4.23)
11. Finally, Density segregation is calculated.

Figure 4.21: Extracting cement samples from particles settling cells

Figure 4.22: Cement pieces

Figure 4.23: Weighting cement samples for density segregation detection

4.11 Shrinkage / Expansion Test

- **Purpose**

To determine cement Shrinkage or expansion phenomena that could take place after cement aging at high pressure and temperature environment.

- **Testing Apparatus**

Shrinkage/Expansion ring molds. (Figure 4.24)

Figure 4.24: Cement expansion/shrinkage mold

- **Procedure**

1. Normal tap water is filled into the mixing jar. (Figure 4.2)
2. Cement powder is mixed with silica products separately. (Figures 4.3 , 4.4)
3. The chemical additives are added to the water in the mixing jar at low mixing speed.
4. The cement powder mixture is then added to the water + chemical additives at mixing speed of 4000 rpm within 3 minutes. (Figure 4.5)
5. The final mixture is then mixed further at 12000 rpm for 35 seconds.(Figure 4.6)
6. The metallic ring molds are prepared to be filled with the slurry. (Figure 4.25)
7. Cement slurry is carefully filled into the ring molds using appropriate syringe. (Figure 4.26)
8. The distance between the ring mold terminals is measured and will be used as the reference for any expansion or shrinkage that could occur after aging. (Figure 4.27)
9. The ring molds are then taken to the HP/HT curing chamber to be conditioned for 168 hours (7 days) at 290 °F temperature and 3000 Psi pressure. (Figure 4.14)
10. After 168 hours, the ring molds are brought out of the curing chamber (Figure 4.28) and distance between the ring mold terminals is measured again and the difference between the two measurements (after aging – before aging) is noted.
11. Positive difference indicates expansion while negative difference indicates shrinkage.

Figure 4.25: Ring molds to be filled with cement slurry

Figure 4.26: Ring mold being filled with cement slurry

Figure 4.27: Ring mold terminals being measured

Figure 4.28: Cement sample after aging for 7 days

(Expansion/shrinkage test)

Chapter 5

Results and Discussion

This chapter presents the results of this study on the effect of Nano silica on Portland Saudi cement type 'G' in high pressure high temperature applications. Earlier in the previous chapter, the experimental program was presented and the well parameters were also introduced. The results of each experimental test are discussed here for the proposed Nano silica percentages of the selected well cement system.

5.1 Cement system validation

Previously in chapter 4, cement systems were introduced in tables (2) and (3) where the only difference between the two designs was the addition of Nano silica in different percentages proposed to be (0, 1, 3, and 5) % by cement weight.

After starting the experimental program, the following observations have been noticed:

- Cement slurry design (Table 2) was easy to implement in the lab and the slurry mixture was easy to condition, test, and clean.
- Cement slurry design (Table 3) was more challenging than the slurry design without the Nano silica addition.

- Presence of Nano silica affected the cement physical properties. "the more the Nano silica added, the more (thixotropic *) the slurry will be"

* Thixotropy is the property of cement slurry to develop high gel after short static period of time.

- Cement slurry design (Table 3) having Nano silica percentage of (1.0%) was successful and selected for further testing.
- Cement slurry design (table 3) having Nano silica percentage of (3.0%) wasn't successful as the slurry materials were difficult to mix (Figure 5.1).

Figure 5.1: Difficulties in mixing the cement slurry having (3.0%) Nano silica

- After several unsuccessful attempts, changes had to be considered to the cement system presented in table (3) for the Nano silica percentage of (3.0%). These changes include increasing the percentage of the dispersant from (0.4%) to (0.8%) and (1.0%). Moreover, silica sand was introduced which has larger particle size and ability to make the slurry thinner (see table 4). The slurry could be mixed, however, was too thick (un-pumpable) to be tested which implies that it reached the maximum limit of Nano silica percentages to be used (Fig.5.2).

Attempt 1	Class G cement powder + 35 % silica flour + 3.0 % Nano silica + 1.0 % Expanding agent + 0.8 % Dispersant + 0.2 % Fluid loss control agent 1 + 0.5 % Fluid loss control agent 2 + 1.0 % Retarder + 0.25 gmDefoamer
Attempt 2	Class G cement powder + 15 % silica flour + 20 % silica sand + 3.0 % Nano silica + 1.0 % Expanding agent + 0.8 % Dispersant + 0.2 % Fluid loss control agent 1 + 0.5 % Fluid loss control agent 2 + 1.0 % Retarder + 0.25 gmDefoamer
Attempt 3	Class G cement powder + 5.0 % silica flour + 30 % silica sand + 3.0 % Nano silica + 1.0 % Expanding agent + 1.0 % Dispersant + 0.2 % Fluid loss control agent 1 + 0.5 % Fluid loss control agent 2 + 1.0 % Retarder + 0.25 gmDefoamer

Table 4: Unsuccessful cement Slurry designs with (3.0 %) Nano silica

Figure 5.2: Unsuccessful cement slurry having (3.0 %) Nano silica

- It was realized that cement system having (3.0%) and (5.0%) Nano silica will not work, thus, Nano silica percentage was reduced to (2.0%).
- Cement slurry design (Table 3) having Nano silica percentage of (2.0%) was thick for testing (Figure 5.3) and needed an increase in the percentage of the dispersant from (0.4%) to (0.6%) and usage of silica sand.

Figure 5.3: Un-pumpable Cement slurry having (2.0%) Nano silica

Therefore, cement slurry design having (2.0%) Nano silica will be as follow:

Class G cement powder + 15 % silica flour + 20 % silica sand + 2.0 % Nano silica + 1.0 % Expanding agent + 0.6 % Dispersant + 0.2 % Fluid loss control agent 1 + 0.5 % Fluid loss control agent 2 + 1.0 % Retarder + 0.25 gmDefoamer	
Slurry Density (Actual), PCF	Unknown
Water to cement Ratio	0.435
Slurry Yield , ft^3/Sack	1.384
Thickening Time (Expected)	Unknown

Table 5: Cement Slurry design with (2.0%) Nano silica

5.2 Effect of Nano silica on cement slurry thickening time

Thickening time is an important property of cement slurry and gives an indication about how long the cement slurry remains pumpable. The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to thickening time test and times of cement slurries to reach a consistency of 100 BC were recorded.

Figures(5.4, 5.5, and 5.6)show the behavior of cement slurries having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) under thickening time test, it can be noticed that the three slurries had consistencies of (31, 40, and 59) BC at the beginning of the test which is an indication of the condition of the slurry viscosities once prepared.

Temperature plays a role in reducing the consistency to some extent at the beginning of the test before it rises again dramatically, and once a consistency of 100 BC is reached, the slurry is considered un-pumpable.

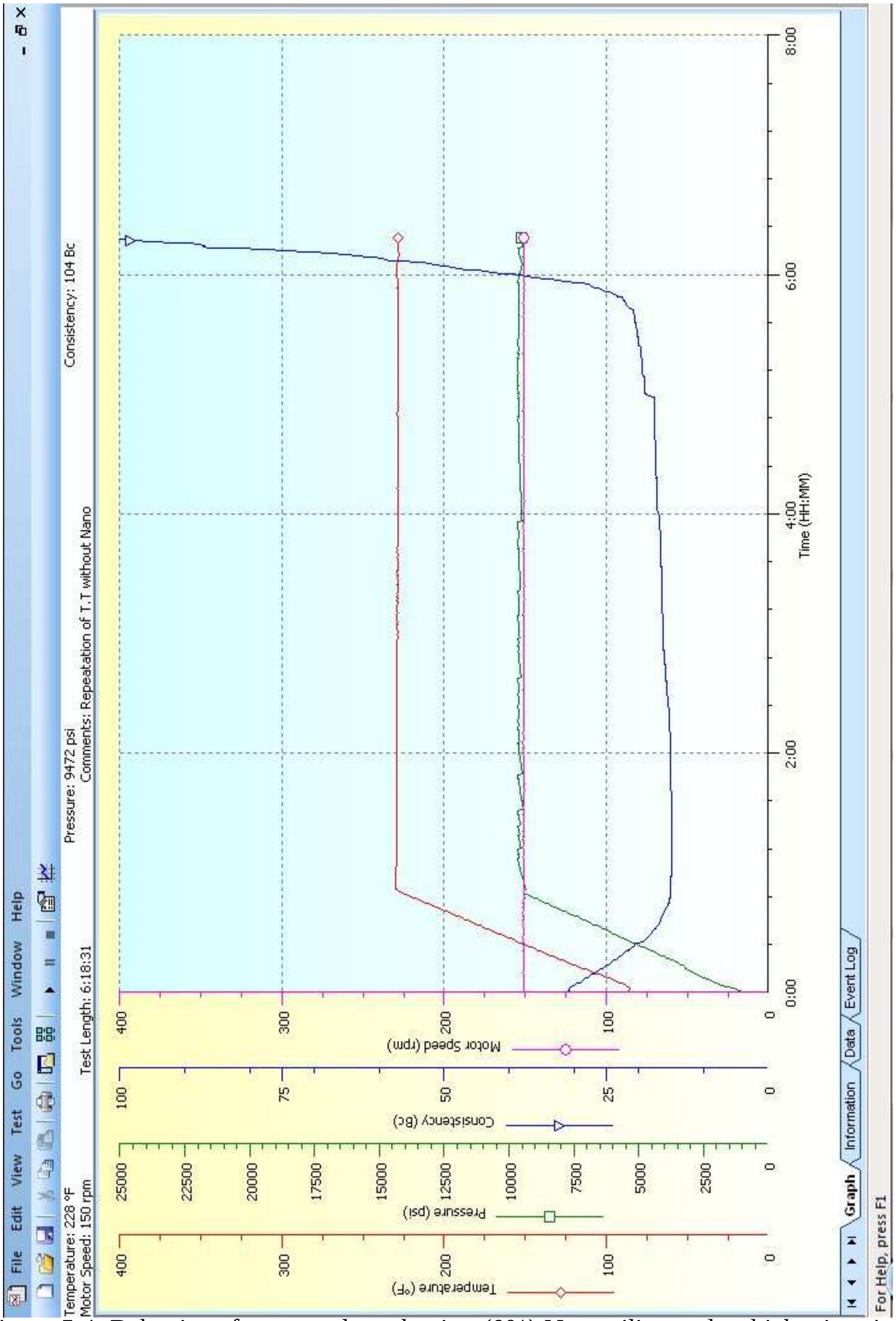


Figure 5.4: Behavior of cement slurry having (0%) Nano silica under thickening time test

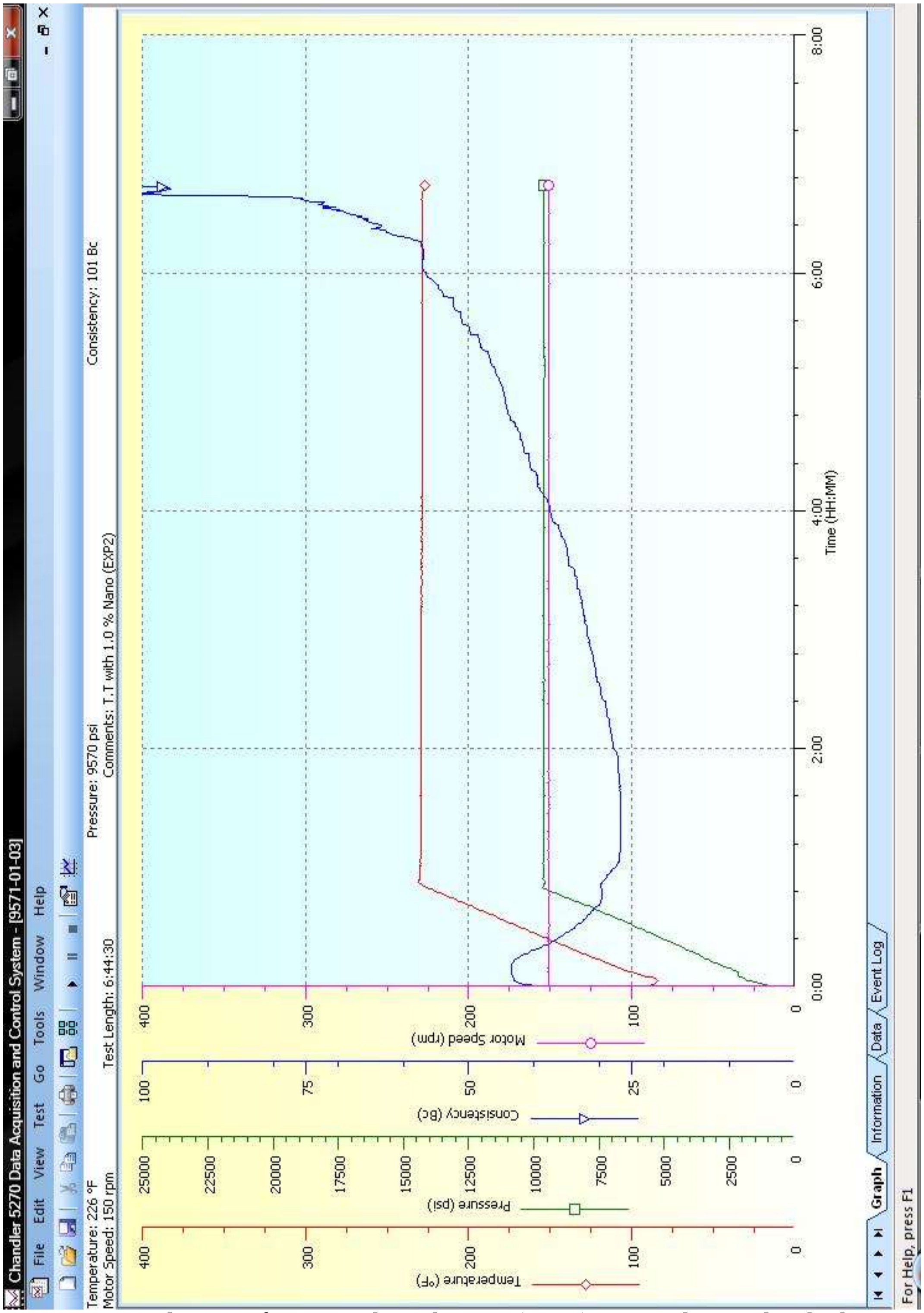


Figure 5.5: Behavior of cement slurry having (1.0%) Nano silica under thickening time test

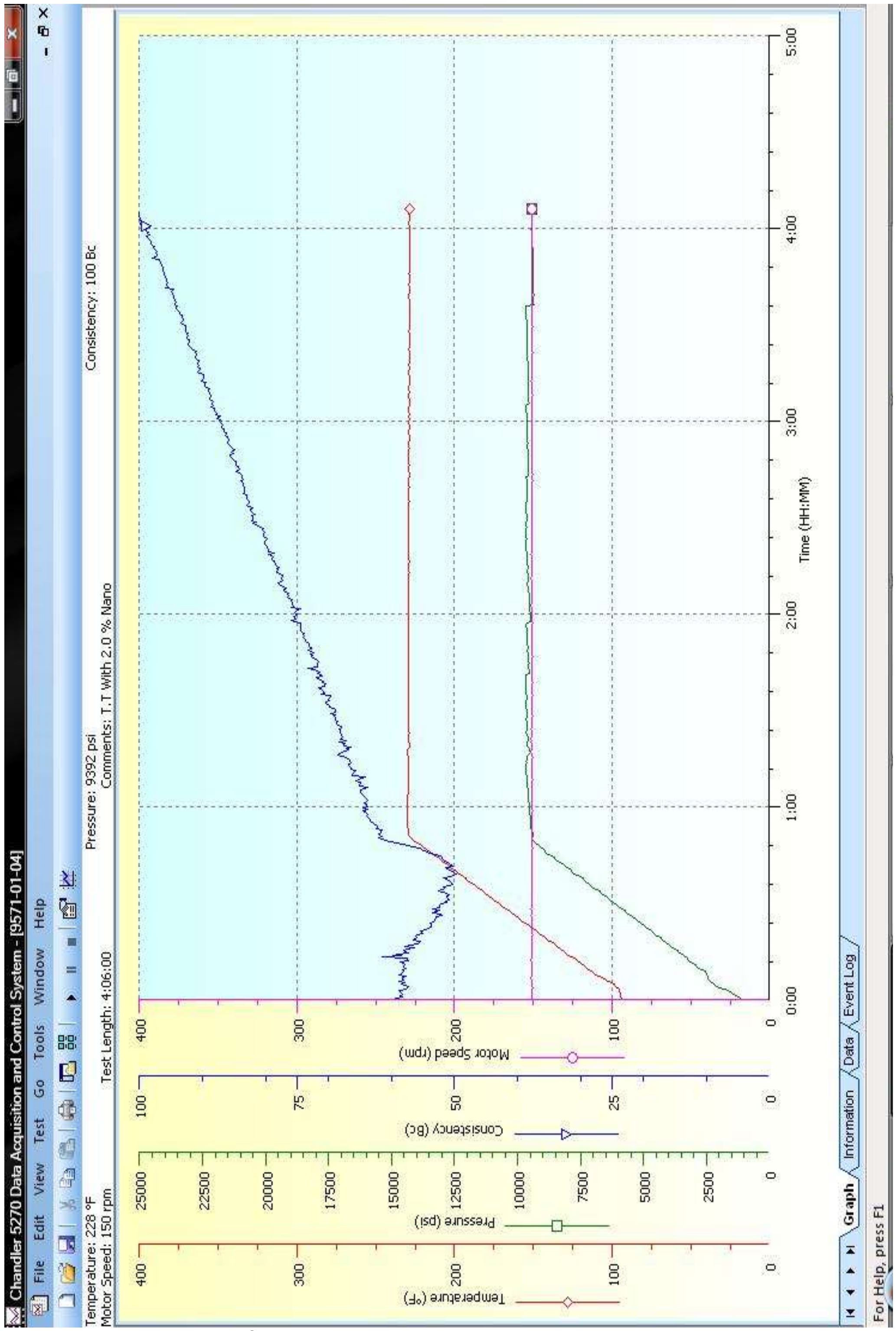


Figure 5.6: Behavior of cement slurry having (2.0%) Nano silica under thickening time test

Figure 5.7 gives a comparison between the thickening times of the cement slurries having Nano silica percentages of (0%, 1.0%, and 2.0%).

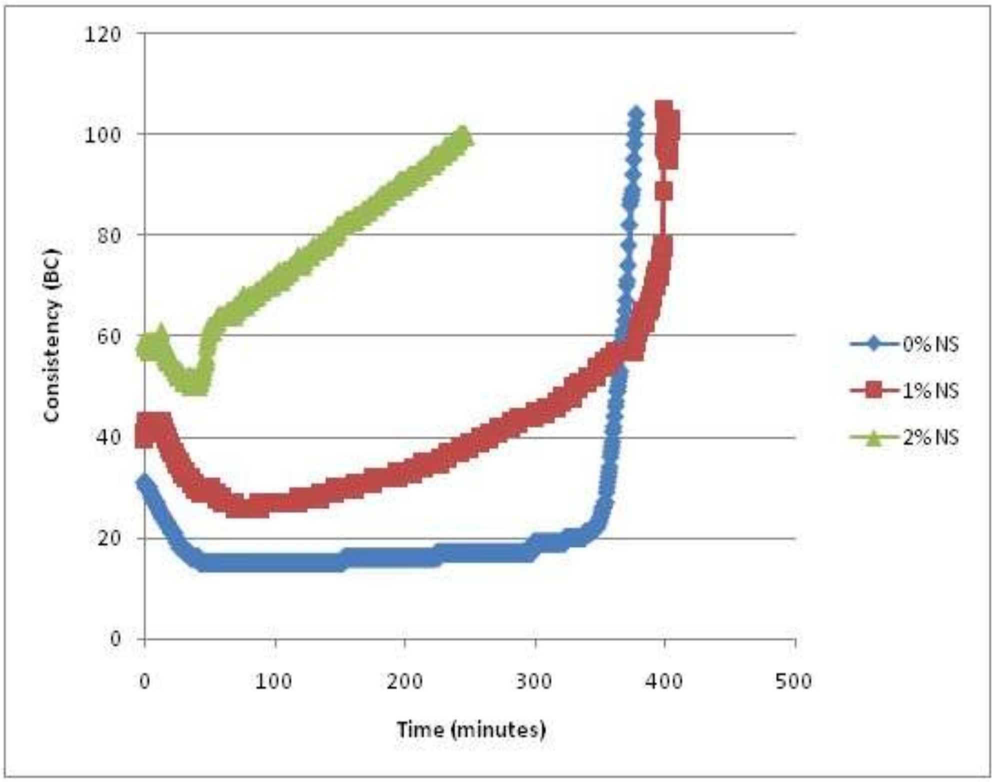


Figure 5.7: Thickening times for the cement slurries having (0, 1, 2) % Nano silica

According to literature [27, 41, 44, 45, 46], Nano silica has the tendency to decrease the thickening time or in other words, accelerating the hydration process of cement slurry. This effect can be observed here as well, in particular, for the cement slurry having Nano silica percentage of (2.0%) where the slurry thickened faster and reached consistency of 100 BC in almost 4 hours while other cement slurries having Nano silica percentage of (0%) and (1.0%) reached consistency of 100 BC in more than 6 hours.

It can be noticed that cement slurry having Nano silica percentage of (1.0%) didn't suffer from any reduction in thickening time. The reason could be due to the presence of silica flour at high percentage (35%) which minimized the effect of Nano silica on the hydration process. However, in cement slurry having Nano silica percentage of (2.0%), silica flour percentage has been reduced to (15%) only and silica sand (20 %) was introduced due to cement slurry preparation concerns. This modification allowed the Nano silica to have an effect on the hydration process.

5.3 Effect of Nano silica on cement slurry fluid loss

Cement slurry fluid loss is another important property to determine how much fluid is lost when the slurry is exposed to a differential pressure. This could occur when cementing across high permeability zones, deep liners, or sensitive formations.

For the well selected in this study, deep liner at depth of 14,000 ft requires cement slurry having an acceptable fluid loss rate. The industry has accepted a fluid loss rate for cementing deep liners in the range of 100 cc/30 min.

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to fluid loss test for 30 minutes under pressure of 1000 psi and temperature of 194 °F.

Table (6) shows the results of the fluid loss test, unlike what have been reported in the literature, Nano silica tended to increase cement slurry fluid loss rate, even though it didn't exceed the acceptable range by industry. The reason could be due to the presence of different material particle sizes (Portland cement, silica flour, silica sand, and Nano silica) which influences the particles bonding and distribution.

Fluid loss rate (cc/30min)	(0%) Nano silica	(1.0%) Nano silica	(2.0%) Nano silica
	36	54	56
	42	63	58
Average (cc/30min)	39	58.5	57

Table 6: Fluid loss rate for the cement slurries having (0, 1, 2) % Nano silica

5.4 Effect of Nano silica on cement slurry free water separation

Water is used in cement slurry design to provide fluidity and to act as a chemical agent in the hydration process. However, if water is used excessively, free water might form on top of the cement while particles settling effect could take place.

To determine the effect of Nano silica on the amount of free water of cement slurry, The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to free water test where they were aged for2 hours under normal room temperature and atmospheric pressure.

The results indicated that the three cement systems showed no water forming and Nano silica didn't introduce any disturbance to the particles suspension in the cement slurry.

Free fluid (%)	(0%) Nano silica	(1.0%) Nano silica	(2.0%) Nano silica
	0	0	0

Table 7: Free water percentages for the cement slurries having (0, 1, 2) % Nano silica

Figure 5.8: Cement slurry under free water test

5.5 Effect of Nano silica on cement slurry rheological properties

Cement rheology is an important factor thatyields in important information which aids in the design of cementing operation. Cement slurry rheological properties measurements give an estimation of the frictional pressure losses and also the pumping power requirement during the cementing operation.

Rheological properties include determining the cement slurry viscosity at different rotational speeds (rpm) and from that plastic viscosity and yield point can be determined.

Viscosity is defined as the resistance of a fluid to flow and is measured as the ratio of shear rate to the shear stress. Plastic viscosity is the slope of shear stress/ shear rate curve while the yield point is the shear stress when the shear rate is zero.

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to rheology test and the results are tabulated below:

Nano Silica (%)	Viscosity(cp) @ 3 rpm	Viscosity(cp) @ 6 rpm	Viscosity(cp) @ 100 rpm	Viscosity(cp) @ 200 rpm	Viscosity(cp) @ 300 rpm
0	5	7	79.5	134.5	209
1	29	35	136	219.5	296
2	79.5	83.5	173	273	300

Table 8: Rheology results for the cement slurries having (0, 1, 2) % Nano silica

Nano Silica (%)	PV (cp)	YP (lbs/100ft²)
0	193.4	8.7
1	216.1	35.4
2	207.3	62.4

Table 9: Plastic viscosity (PV) and Yield point (YP) for the cement slurries having (0, 1, 2) % Nano silica

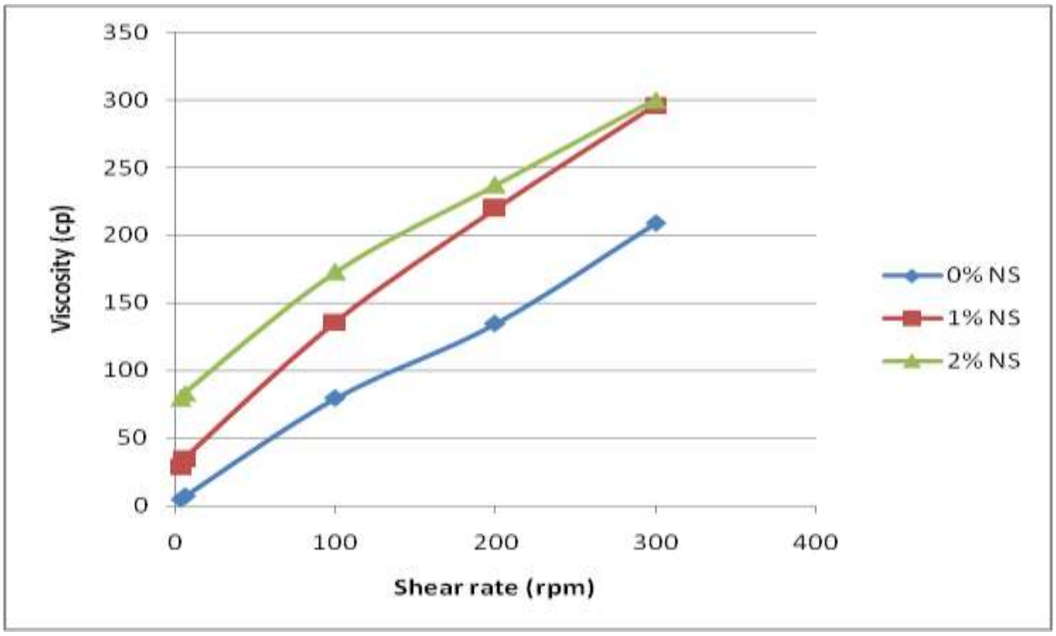


Figure 5.9: Shear rate / shear stress diagram

Figure (5.9) and table (9) shows clearly the effect of Nano silica on the rheological properties of cement slurry as both plastic viscosity and yield point increased with the increase of Nano silica percentage added to the cement slurry.

This fact can make the Nano silica act as a viscofier agent, however it should be used with caution since Nano silica affects other cement slurry properties as well.

5.6 Effect of Nano silica on cement compressive strength

Cement compressive strength is a major consideration when performing cementing operations. Pumping the cement efficiently, placing it safely on time, assuring cement integrity after placement (prior to resuming drilling operation) are all issues to be considered. Therefore, compressive strength tests are conducted to obtain an estimation of the development of cement strength with time utilizing the ultrasonic cement analyzer (UCA) and also to determine cement bonding stability after set utilizing the conventional compressive strength test (Crushing).

5.6.1 Compressive strength by "crushing"

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to the API compressive strength where 6 cement cubes have been prepared for each cement system. The cubes are subjected to an axial increasing load in the compressive strength tester until they crack (figure 5.10).

Figure 5.10: Cement sample having (0%) Nano silica under compressive strength test

For more cement samples crushing graphs, kindly refer to the appendix.

The following table summarizes the results of the compressive strength test.

	Sample No.	(0%) Nano Silica	(1.0%) Nano Silica	(2.0%) Nano Silica
Compressive strength (Psi)	1	2979	2358	2639
	2	3470	2815	5530
	3	2877	2965	3252
	4	2790	2571	2897
	5	2842	2080	5095
	6	3279	2254	3704
Average		**3039 Psi**	**2507 Psi**	**3853 Psi**

Table 10: Compressive strength values for the cement samples having (0, 1, 2) % Nano silica

Compressive strength values in table (10) showed inconsistency, since each cement system has a range of compressive strength values. Averagely speaking, all three cement systems met industry requirement of achieving a minimum compressive strength of 500 psi needed after cement set, and prior to resuming the drilling operation. Moreover, all three cement systems met industry requirement of achieving a minimum strength of 2000 psi needed to withstand perforation shots without causing damage to the cement.

According to literature, Nano silica tends to increase the compressive strength of cement and this was clear in the cement system having Nano silica percentage of (2.0 %) since it yielded in high compressive strength values compared to the other two cement systems.

5.6.2 Compressive strength by ''sonic waves''

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to the ultra sonic cement analyzer (UCA) test under high temperature (290 °F) and pressure (4666 Psi) for 24 hours.

Figures (5.11, 5.12, and 5.13) show the development of compressive strength with time for the three cement systems.

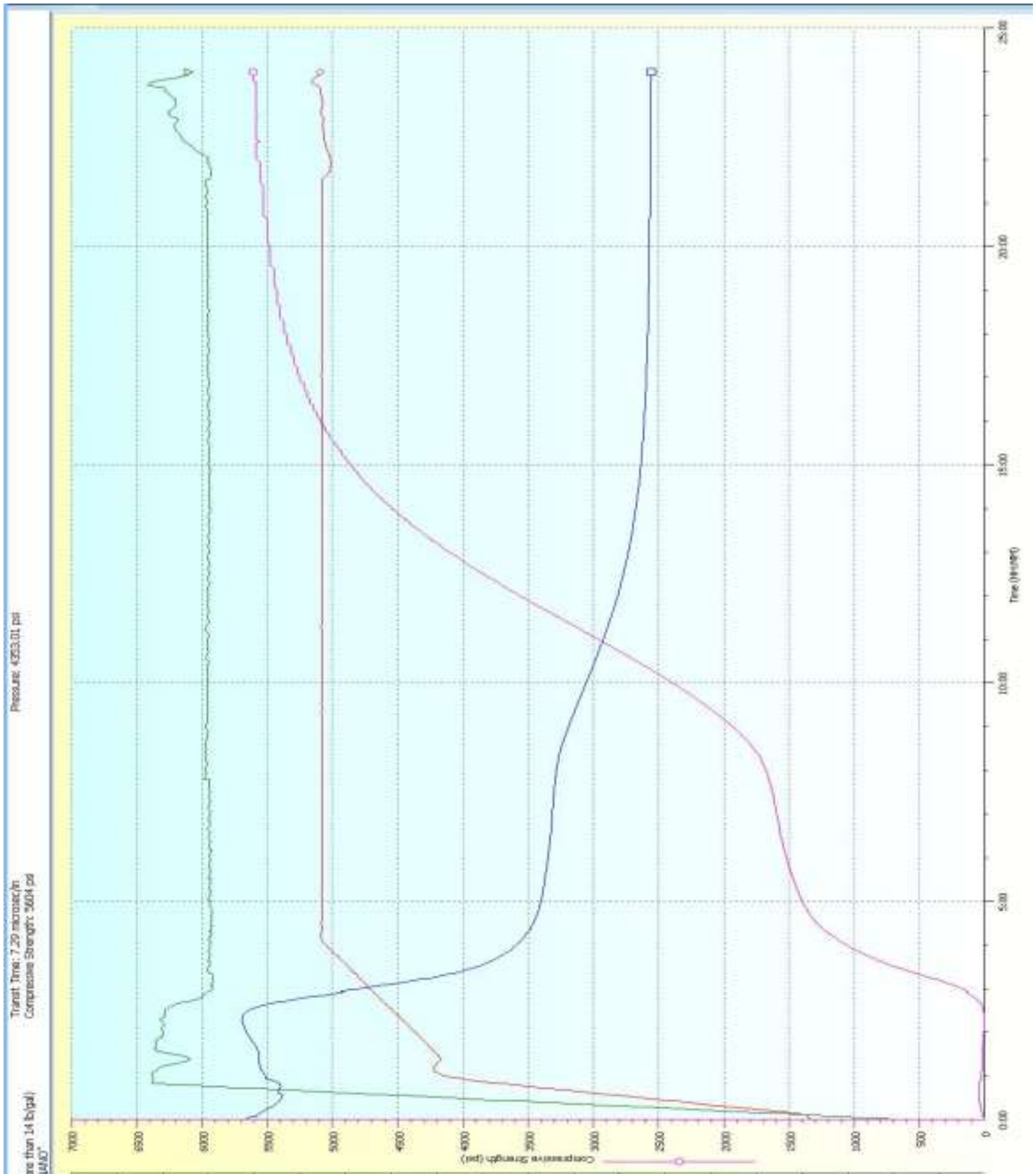


Figure 5.11: Compressive strength development with time for cement slurry having (0%) Nano silica

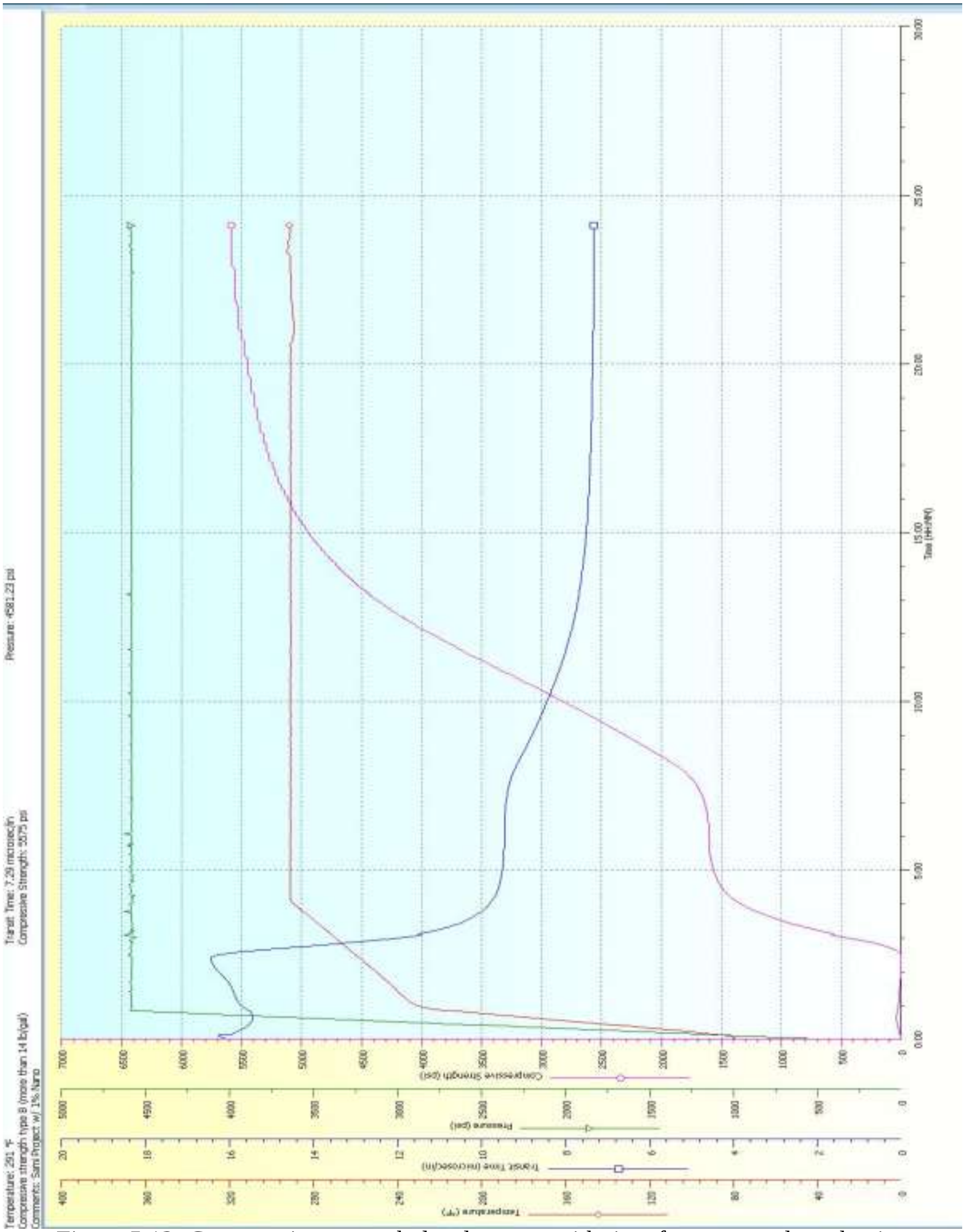


Figure 5.12: Compressive strength development with time for cement slurry having (1.0%) Nano silica

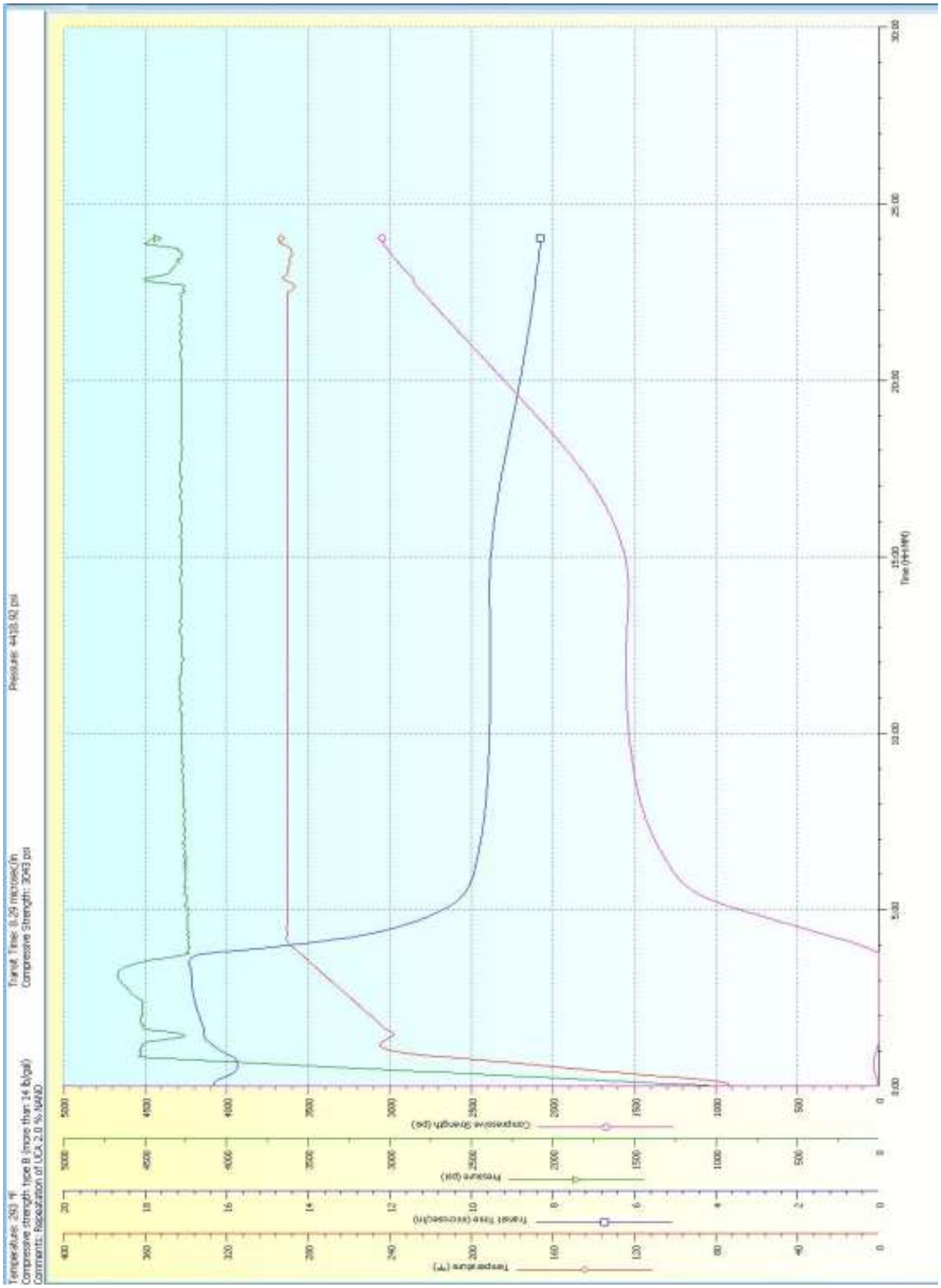


Figure 5.13: Compressive strength development with time for cement slurry having (2.0%) Nano silica

Figure 5.14 shows the performance of the three cement systems under UCA test where cement slurry having Nano silica percentages of (0%) and (1.0%) developed similar compressive strength trends which confirm the dominance of silica flour (35 %) that minimized Nano silica effect. However, cement slurry having Nano silica percentage of (2.0%) developed lower compressive strength trend (unlike what was obtained by the crash test). The reason for this might be due to the UCA operating mechanism where the sonic waves travels between the two cell terminals in a certain transit time which is then correlated to a corresponding compressive strength value. Knowing that, cement slurry having Nano silica percentage of (2.0%) consists of different material particle sizes (Portland cement, silica flour, silica sand, and Nano silica) which influences the path of the sonic wave and consequently the compressive strength obtained from the transit time.

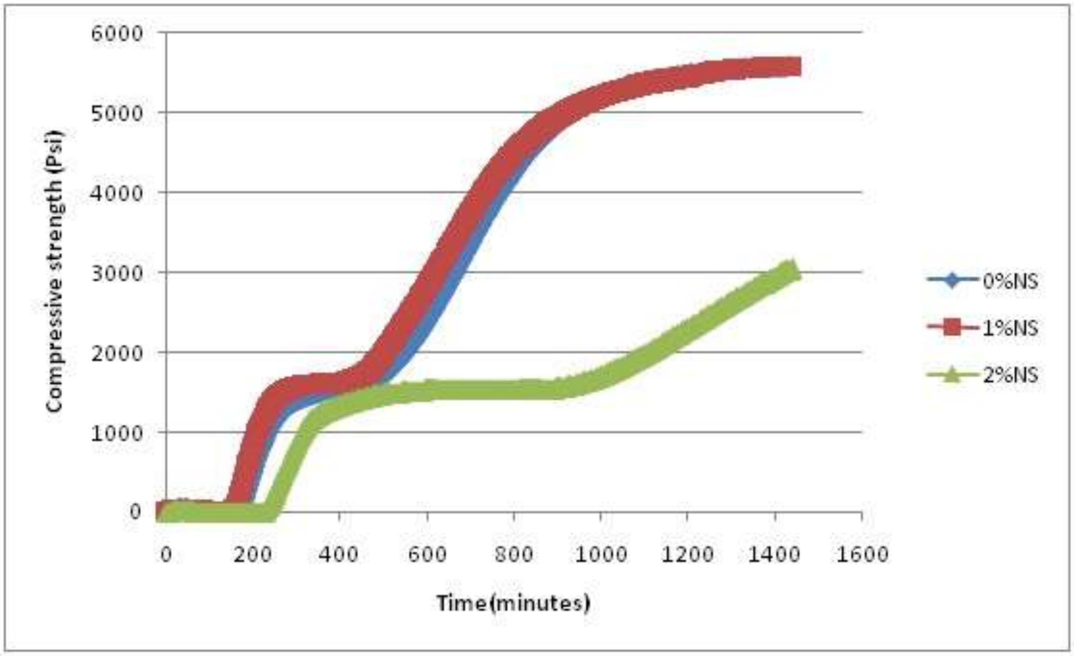


Figure 5.14: Compressive strength development with time for the cement slurries having (0, 1, 2) % Nano silica

Table (11) and figure (5.15) represent the cement slurry development with time and focus on the time required for each cement system to develop a compressive strength of 50 Psi and 500 psi. These compressive strengths are considered sufficient enough to support the steel casing / liner prior to resuming the drilling operation. The transition period between developing a compressive strength of 50 psi and 500 psi is important and needed to be as short as possible to avoid long waiting time on cement before resuming drilling operation. Cement slurry having Nano silica percentage of (1.0%) yielded in the shortest transition period (23 minutes) while the cement slurry having Nano silica percentage of (0%) and (2.0%) yielded on transition periods of (35 and 38) minutes respectively.

	(0%) Nano silica	(1.0%) Nano silica	(2.0%) Nano silica
50 Psi (minutes)	164	158	233
500 psi (minutes)	199	181	271
24 hours strength (Psi)	5604	5575	3043

Table 11: Development of compressive strength with time for the cement samples having (0, 1, 2) % Nano silica

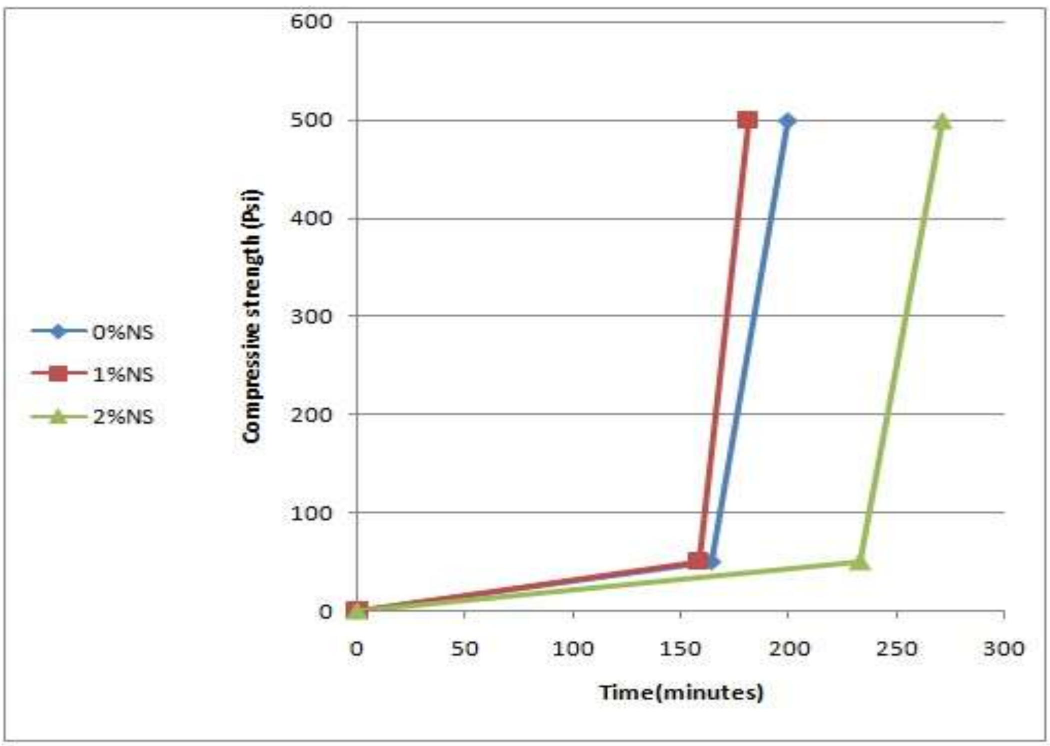


Figure 5.15: Time required to reach 50 and 500 (Psi) compressive strength for the cement slurries having (0, 1, 2) % Nano silica

It should be noted that compressive strength values obtained from the UCA may not agree to the ones obtained from crash test which is the case in this study. Crash test yields in more realistic results; however it doesn't represent downhole conditions in the sense of confining pressure and temperature. UCA does subject the cement to high pressure and temperature, but the fact that compressive strength obtained from UCA is correlated out of the transit time measurements; makes it under uncertainties. Therefore, compressive strength values obtained from the UCA must be used with caution.

5.7 Effect of Nano silica on cement static gel strength

Cement slurry static gel strength is an important property to be considered when performing cementing operation. Static gel strength is analyzed experimentally utilizing the static gel strength analyzer (SGSA).

SGSA test reveals the static gel strength development process with time in which cement slurry transition period is obtained. The transition period is defined as the period in which the slurry will develop a gel strength from 100 ($lbs/100ft^2$) to 500 ($lbs/100ft^2$) and will start changing from fluid state into solid state. This transition is critical and must occur when the slurry has reached its destination (e.g. behind the casing / liner) to prevent any possibility for gas migration.

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to the SGSA test under high temperature (290 °F) and pressure (4666 Psi) for 24 hours.

Figure 5.16 shows the static gel strength development process for the three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %). It is evident that Nano silica has a direct effect on the static gel strength since it caused a delay in the gellation process. Moreover, table (12) and figure (5.17) show another effect of Nano silica in terms of reduction in the transition period of cement slurry having Nano silica percentage of (2.0 %).

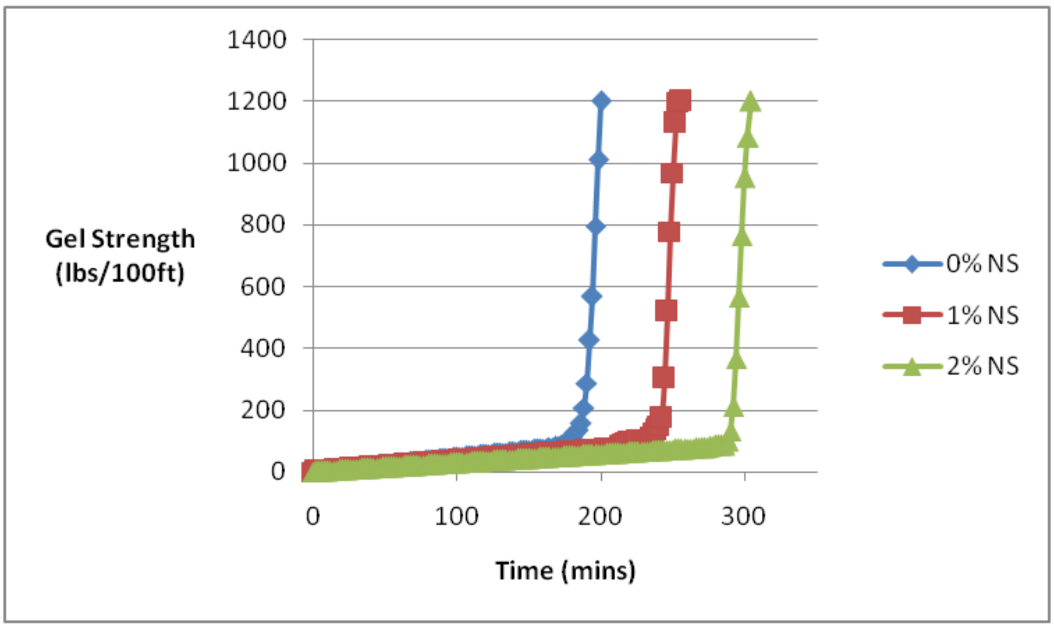


Figure 5.16: Static gel strength development with time for the cement slurries having (0, 1, 2) % Nano silica

Nano silica (%)	100 (lbs/100ft²) Gel Strength (minutes)	500 (lbs/100ft²) Gel Strength (minutes)	Transition period (minutes)
0	176.6	193.1	16.5
1	228	245.8	17.8
2	288.1	295.4	7.3

Table 12: Development of compressive strength with time for the cement samples having (0, 1, 2) % Nano silica

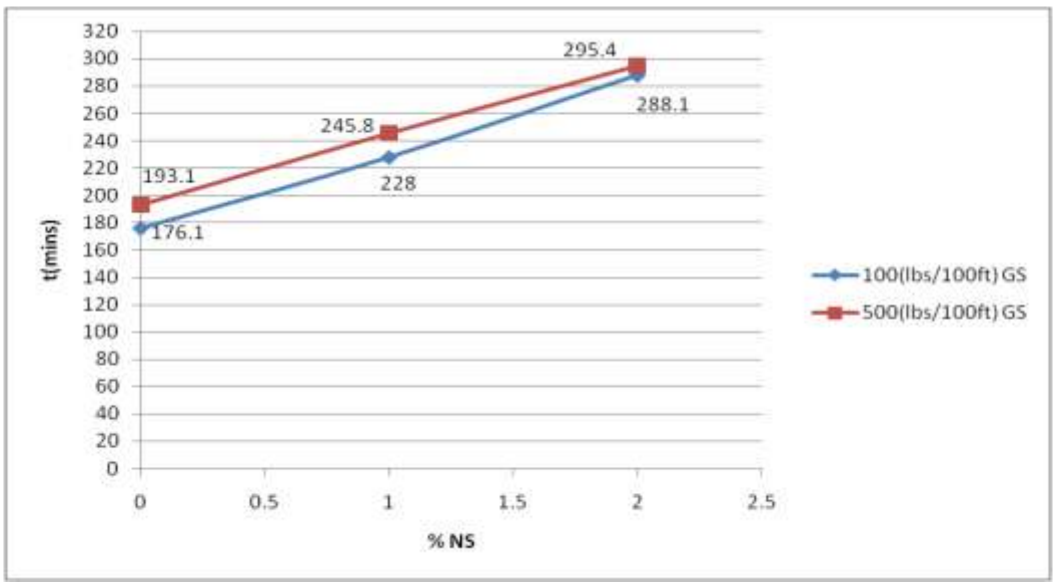


Figure 5.17: Time required to reach 100 and 500 (lbs/100ft²) gel strength for the cement slurries having (0, 1, 2) % Nano silica

5.8 Effect of Nano silica on cement slurry density

Cement slurry density is important for well control consideration. The designed cement slurry density for the selected well is 125 Pcf. However the actual density needs to be measured in the lab utilizing the pressurized density balance.

The three cement system densities having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been measured in the lab and the results are tabulated below.

Nano silica (%)	ρ_{design} (lbs/ft³)	$\rho_{measured}$ (lbs/ft³)
0	125	127
1	125	127
2	125	125.5

Table 13: Densities for the cement samples having (0, 1, 2) % Nano silica

According to literature, Silica is considered as a lightening agent and Nano silica have been reported to decrease slurry density. This wasn't evident in this study since the cement slurries having Nano silica percentages of (0%) and (1.0%) didn't suffer from density degradation due to the presence of silica flour (35%) which again minimized the effect of Nano silica. On the other hand, the effect on cement slurry having Nano silica percentage of (2.0%) was minimal.

It should be noted that density values reported here are subjected to human measurement skills as well as density balance calibration accuracy.

5.9 Effect of Nano silica on cement particles settling

Cement particles settling is a phenomena that may occur in the cement after placement. Density segregation across the cement column is a consequence of the particles settling which is influenced by the suspension property of cement slurry.

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to particles settling test where the cement sample is cut into 3 pieces (top, middle, and bottom). The test results are shown in table (14).

Nano silica (%)	**Density (Pcf)**
(0%) [Top]	125.4
(0%) [Middle]	127.2
(0%) [Bottom]	132.2
(1.0%) [Top]	128.0
(1.0%) [Middle]	127.5
(1.0%) [Bottom]	128.0
(2.0%) [Top]	128.4
(2.0%) [Middle]	128.0
(2.0%) [Bottom]	128.1

Table 14: Densities for different cement sections having (0, 1, 2) % Nano silica

The results indicated that density segregation took place in the cement sample having Nano silica percentage of (0%) since density difference was noted from top (125.4 Pcf) to bottom (132.2 Pcf) of the cement sample. In the other hand, the other two cement samples having Nano silica percentages of (1.0%) and (2.0%) exhibited good suspension capability for the cement sample particles.

5.10 Effect of Nano silica on cement Shrinkage/Expansion

Cement expansion/shrinkage is a major consideration after cement placement. It is desirable to have cement expansion in the annular space between the steel casing/liner and the formation to allow the cement slurry to fill the small voids in the formation and to prevent any path that might be created in the case of cement causing fluid movement behind casing or gas migration behind liner.

The three cement systems having Nano silica percentages of (0 %, 1.0 %, and 2.0 %) have been subjected to cement shrinkage/expansion test for 168 hours (7 days) at 290 (°F) temperatures and 3000 (Psi) pressure.

Radial expansions are calculated for each cement sample and shown in Table (15).

Nano silica (%)	Radial Expansion (%)
0	3.44
1	2.39
2	2.75

Table 15: Expansion of the cement samples having (0, 1, 2) % Nano silica

All cement samples exhibited expansion after 7 days of aging under temperature and pressure indicating that Nano silica didn't cause shrinkage to the cement or adverse effect on the expanding additive.

It should be noted that the three cement systems had the same amount of expanding additive (1.0%).

Chapter 6

Conclusion and Recommendation

6.1 Conclusion

This thesis aimed to investigate the effect of Nano silica on Portland Saudi cement type 'G' in high pressure high temperature applications. Several cement tests have been carried out for this purpose to cover different cement properties such as thickening time, fluid loss, free water separation, rheological properties, compressive strength, static gel strength, slurry density, particles settling, and shrinkage/expansion.

The results of this study are limited to the selected well specifications, cement used, chemical additives, percentages of Nano silica, and cement slurry preparation and testing procedures. However, this study would be useful for researchers interested in this area and would provide valuable information about cement testing techniques and analysis.

Conclusions reached in this study are summarized below:

- It was evident from the thickening time test that Nano silica acts as an accelerator agent to the cement hydration process since it shortened the thickening time for the cement slurry having Nano silica percentage of (2.0%).

- The fluid loss test indicated that Nano silica tends to increase cement slurry fluid loss rate when mixed with other silica products such as silica flour and silica sand. This is due to the presence of different particles sizes in the cement slurry.

- The free water test showed that Nano silica didn't cause any free water separation from the cement slurry column after aging. This was noticed in all Nano silica percentages used (0%, 1.0%, and 2.0%).

- The Rheology test proved the ability of Nano silica to increase cement slurry viscosity as the Nano silica percentage increases. This fact can make the Nano silica act as a viscofier agent; however it should be used with caution since excessive increase in Nano silica percentage results in negative effects (Un-pumpable slurry).

- The compressive strength test "crushing" showed a growth in the average compressive strength of the cement samples having Nano silica percentages of (2.0%).

- The compressive strength test "sonic waves" revealed the ability of Nano silica to develop compressive strength faster. This was noticeable in cement sample having Nano silica percentage of (1.0%).

- The static gel strength test showed that Nano silica has a direct effect on the static gel strength since it caused a delay in the gellation process for cement slurry having Nano silica percentage of (1.0% and 2.0%). Moreover Nano silica reduced the transition period of cement slurry having Nano silica percentage of (2.0 %).

- It wasn't evident from the density test that the Nano silica has a great impact on slurry density since the cement slurries having Nano silica percentages of (0%) and (1.0%) didn't suffer from density degradation and the effect on slurry having Nano silica percentage of (2.0%) was minimal.

- Particles settling test exhibited the ability of Nano silica to suspend cement particles which prevents density segregation across cement column.

- Cement shrinkage/expansion test indicated that Nano silica didn't cause shrinkage to the cement or adverse effect on the expanding additive.

- Presence of silica flour at high percentage in the cement minimized the effect of Nano silica in some tests. While the presence of different material particle sizes (Cement powder, silica sand, silica flour and Nano silica) affected the tests (UCA and SGSA) that rely on sonic waves' transit time.

- Nano silica percentages higher than (2.0%) couldn't be achieved due to difficulties in mixing cement slurry materials.

6.2 Recommendations

The following recommendation might be adopted for future researches

- Investigating the effect of Nano silica on cement was influenced by the presence of silica flour and silica sand. Therefore, excluding other silica products and using Nano silica alone could give Nano silica better advantage to improve cement system performance.

- Investigating the effect of Nano silica on cement in other shallower application (e.g. intermediate casing) might be considered with lower pressure and temperature and lighter cement slurry with different water to cement ratios.

- Scanning Electron Microscope (SEM) is a useful technique to get magnified images which could be used to understand and analyze in depth the particles distribution and bonding before and after adding Nano silica mixed with other micro silica products such as silica flour and silica sand.

- Other techniques including X-ray fluorescence (XRF) and X-ray Diffraction (XRD) are also useful to reveal the mineralogy and composition of cement systems having Nano silica.

Appendix: Compressive Strength samples "Crushing"

Figure A.1: Cement sample having 0 % Nano silica under compressive strength test

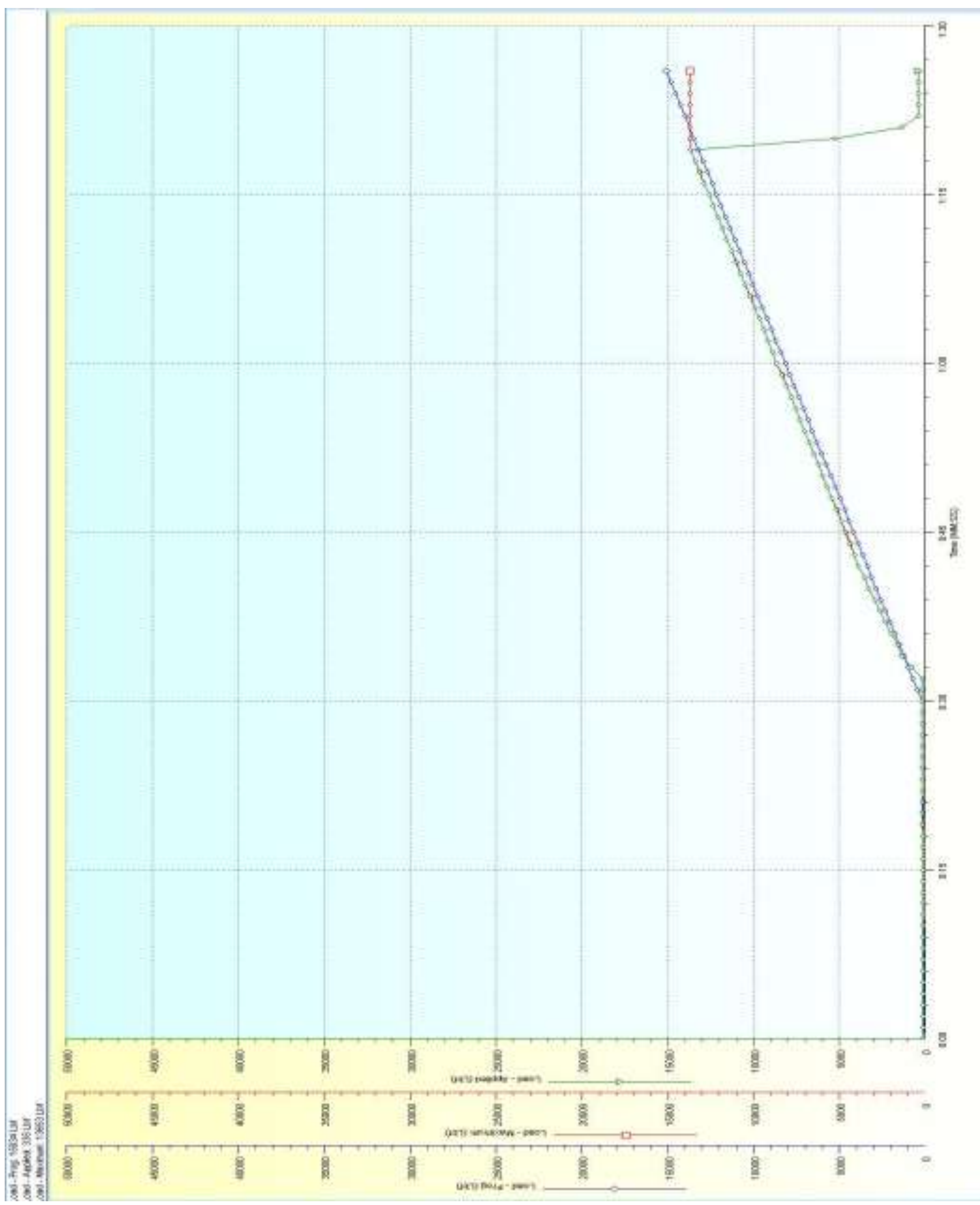


Figure A.2: Cement sample having 0 % Nano silica under compressive strength test

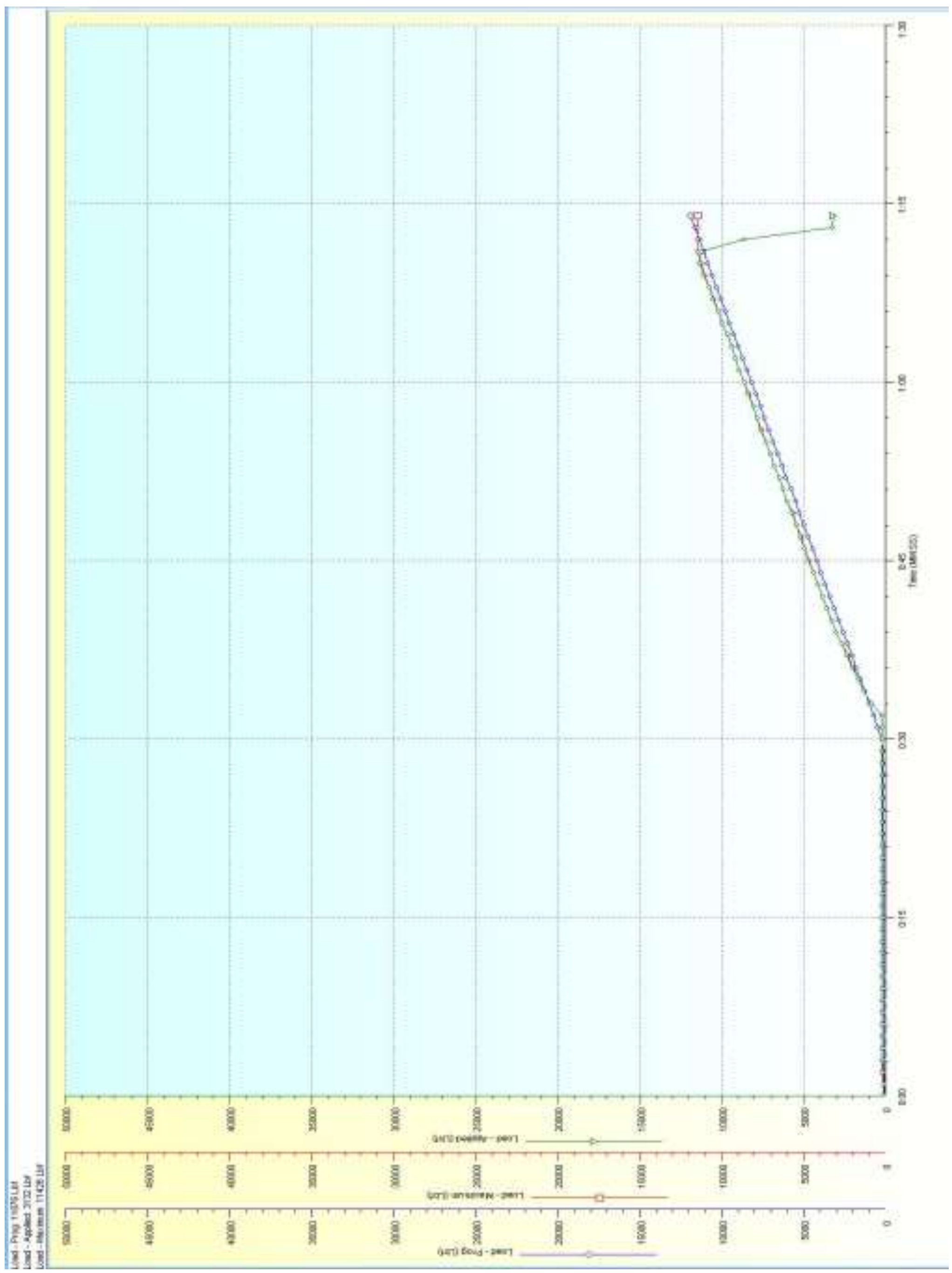


Figure A.3: Cement sample having 1.0 % Nano silica under compressive strength test

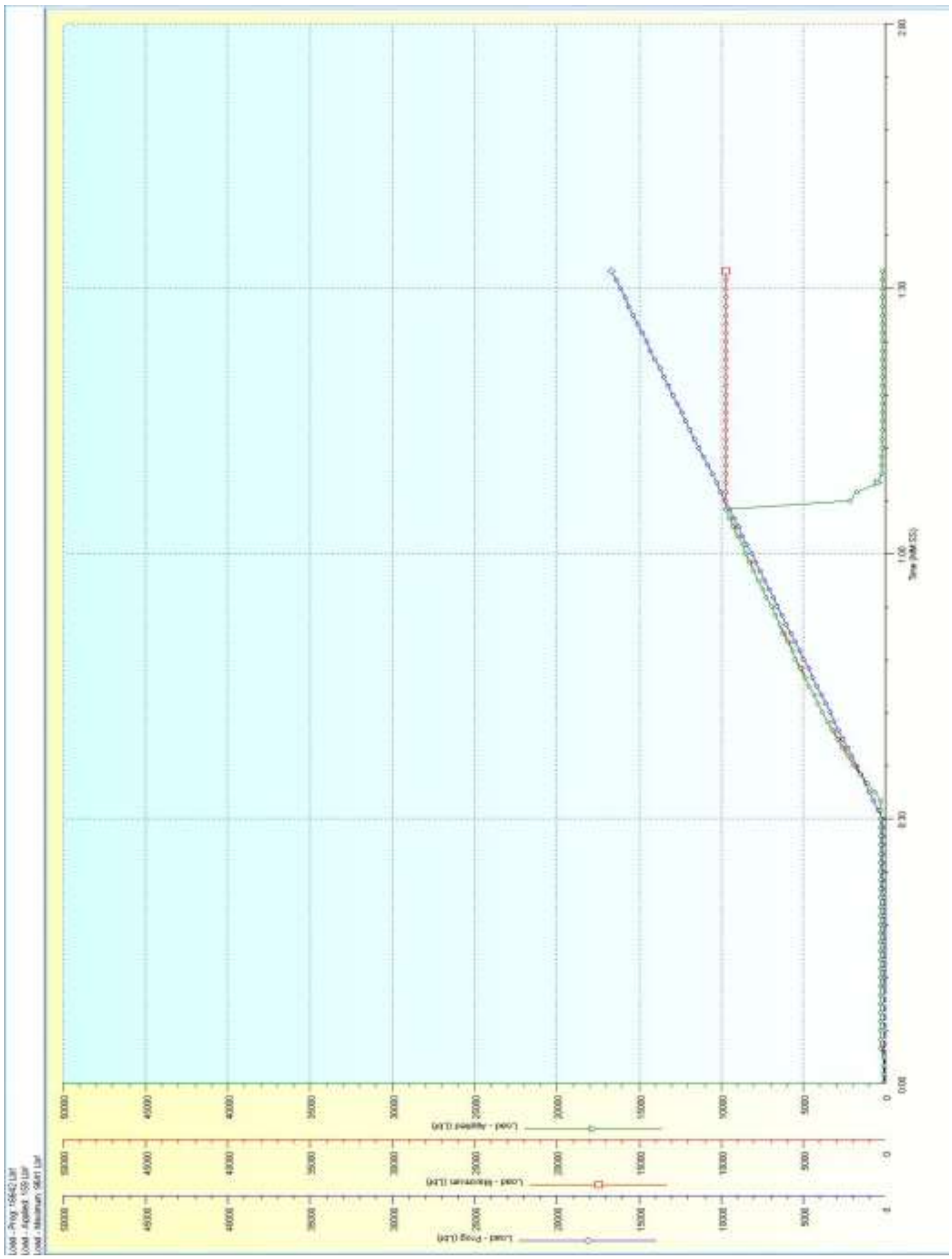


Figure A.4: Cement sample having 1.0 % Nano silica under compressive strength test

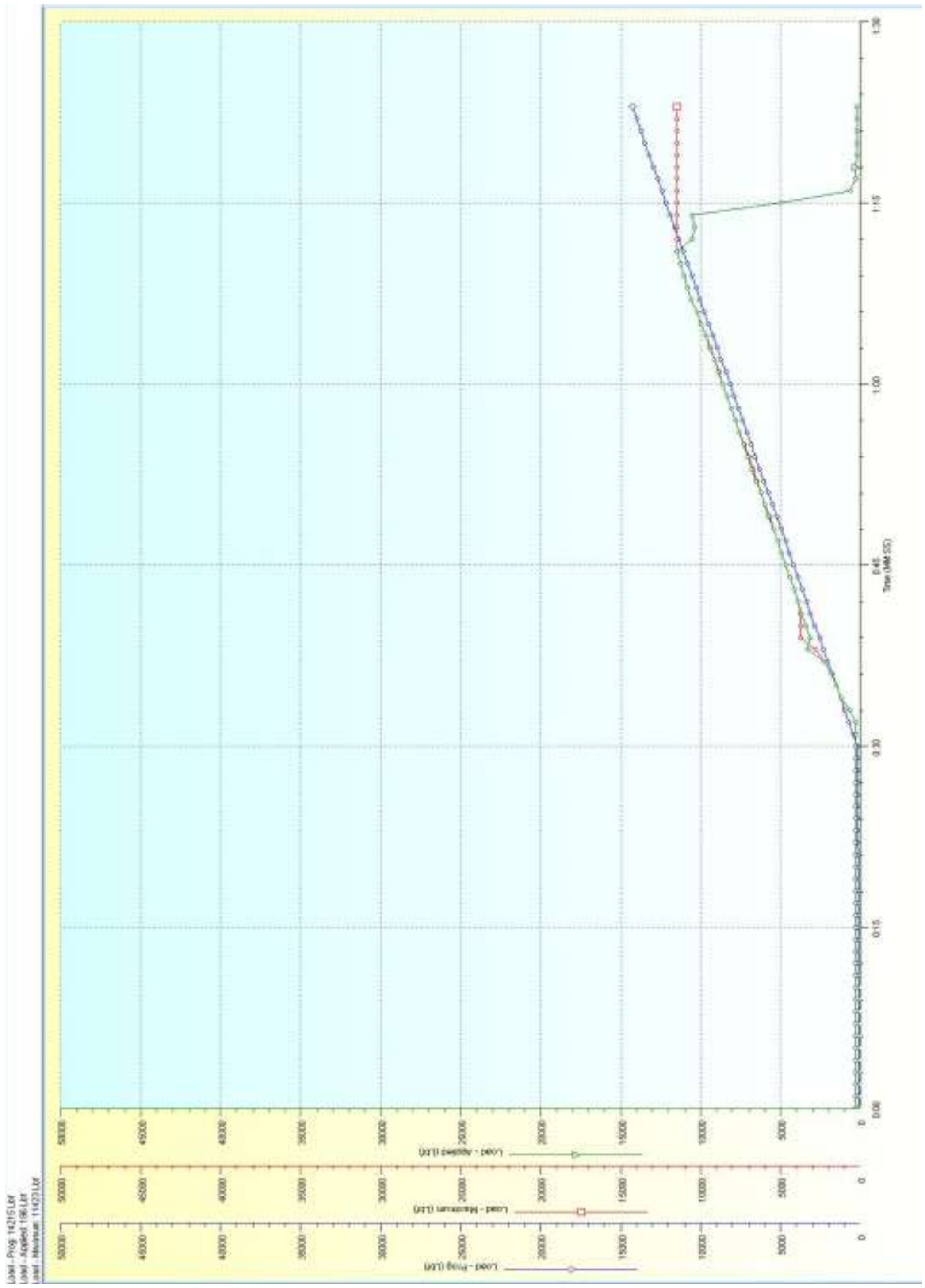


Figure A.5: Cement sample having 1.0 % Nano silica under compressive strength test

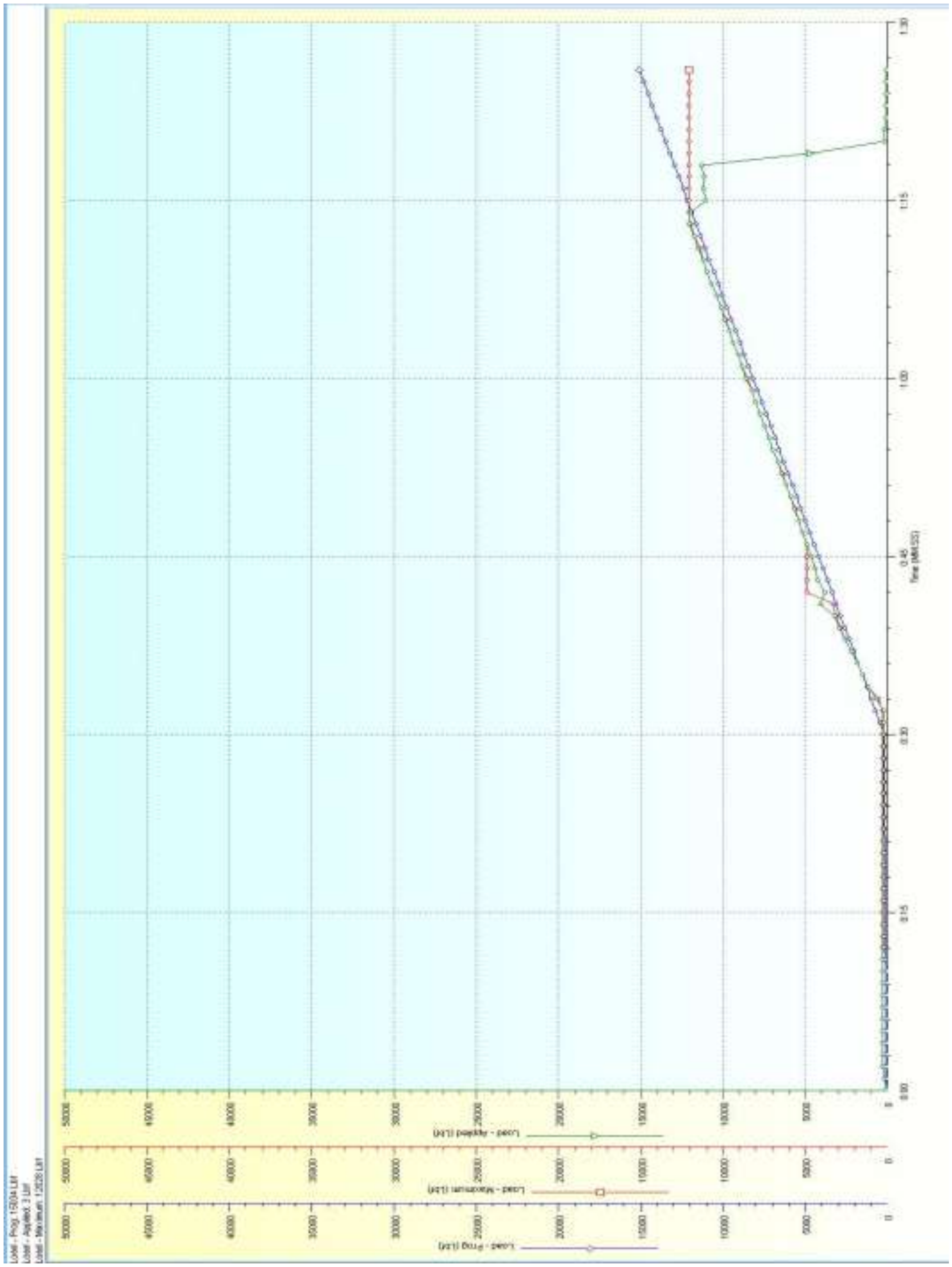


Figure A.6: Cement sample having 2.0 % Nano silica under compressive strength test

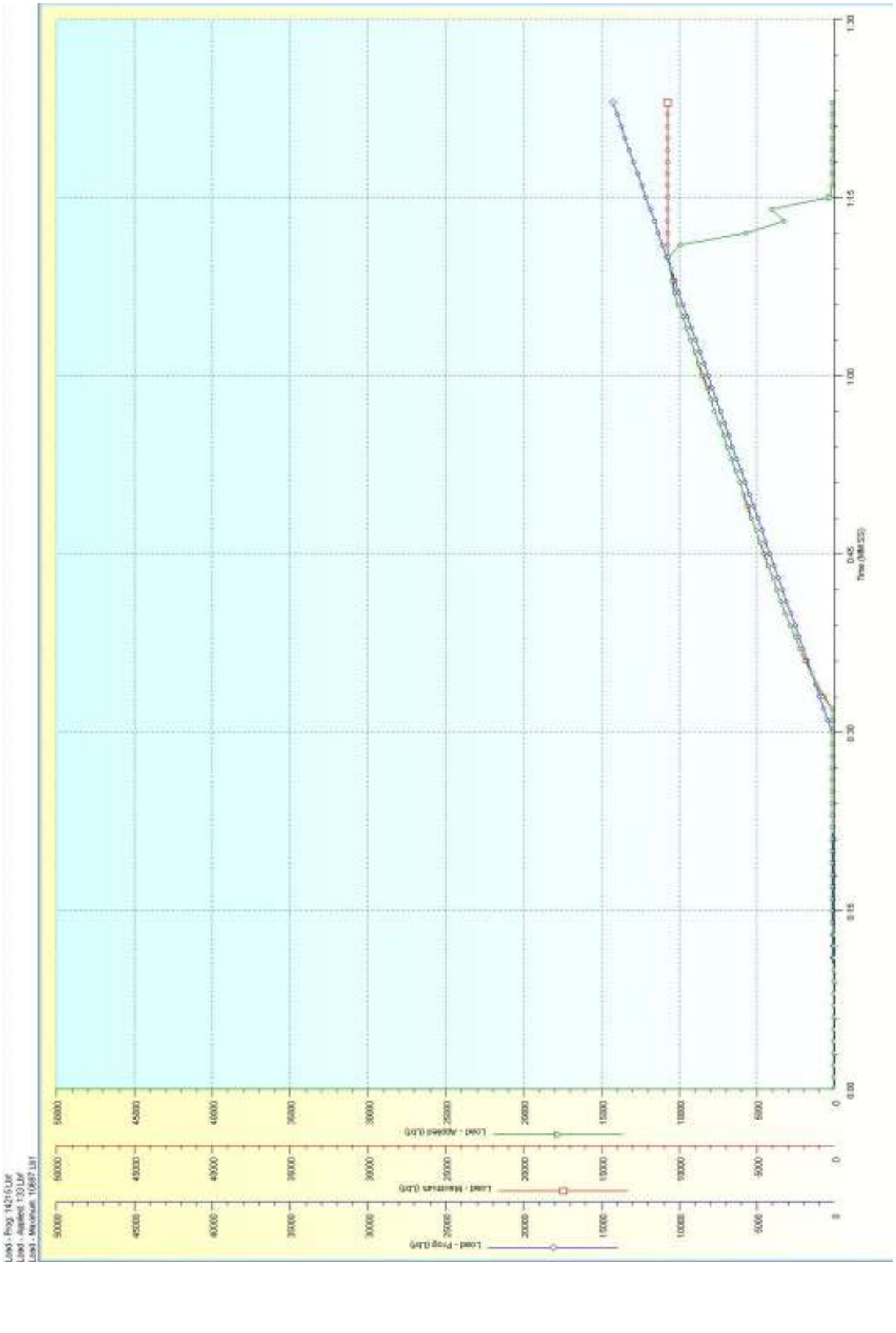


Figure A.7: Cement sample having 2.0 % Nano silica under compressive strength test

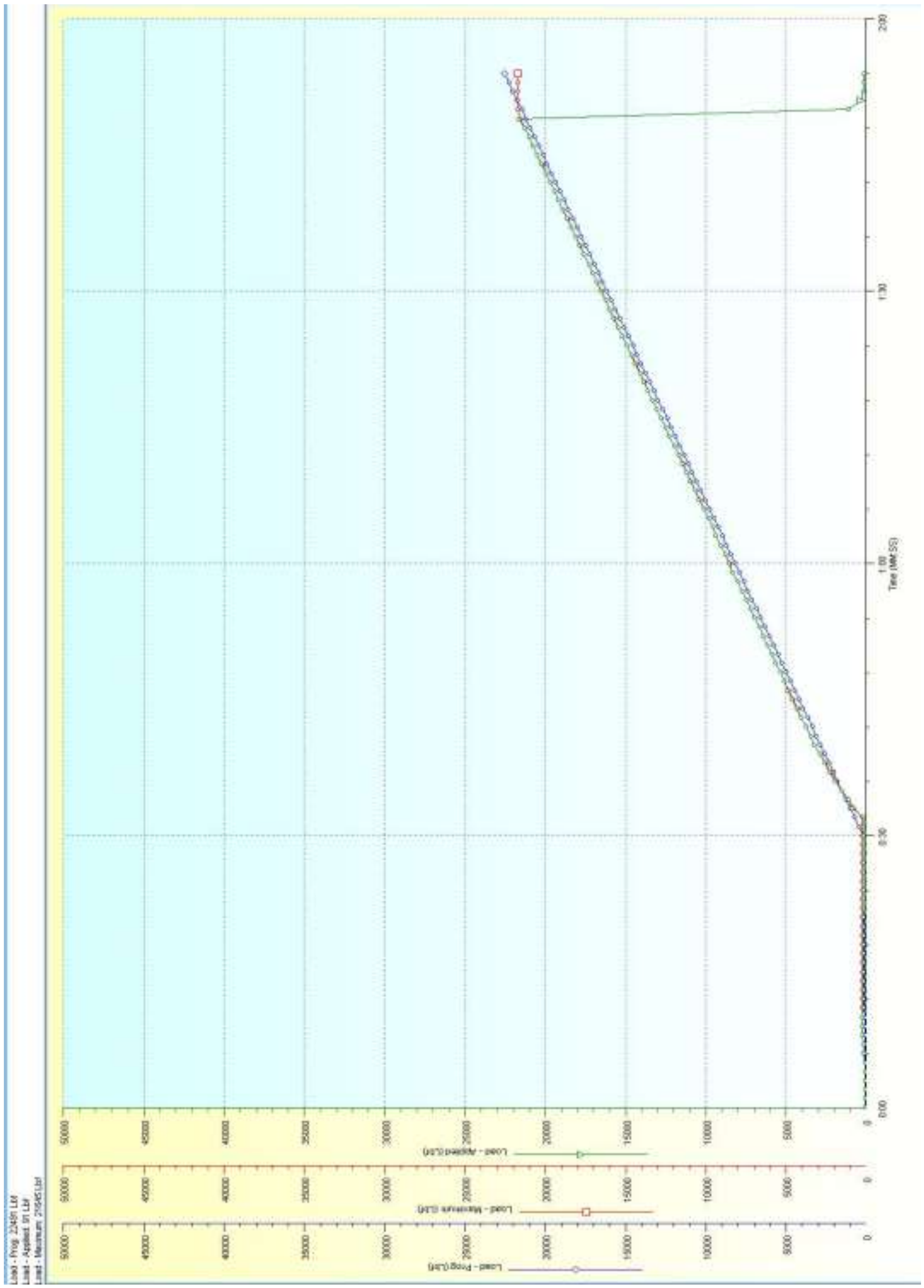
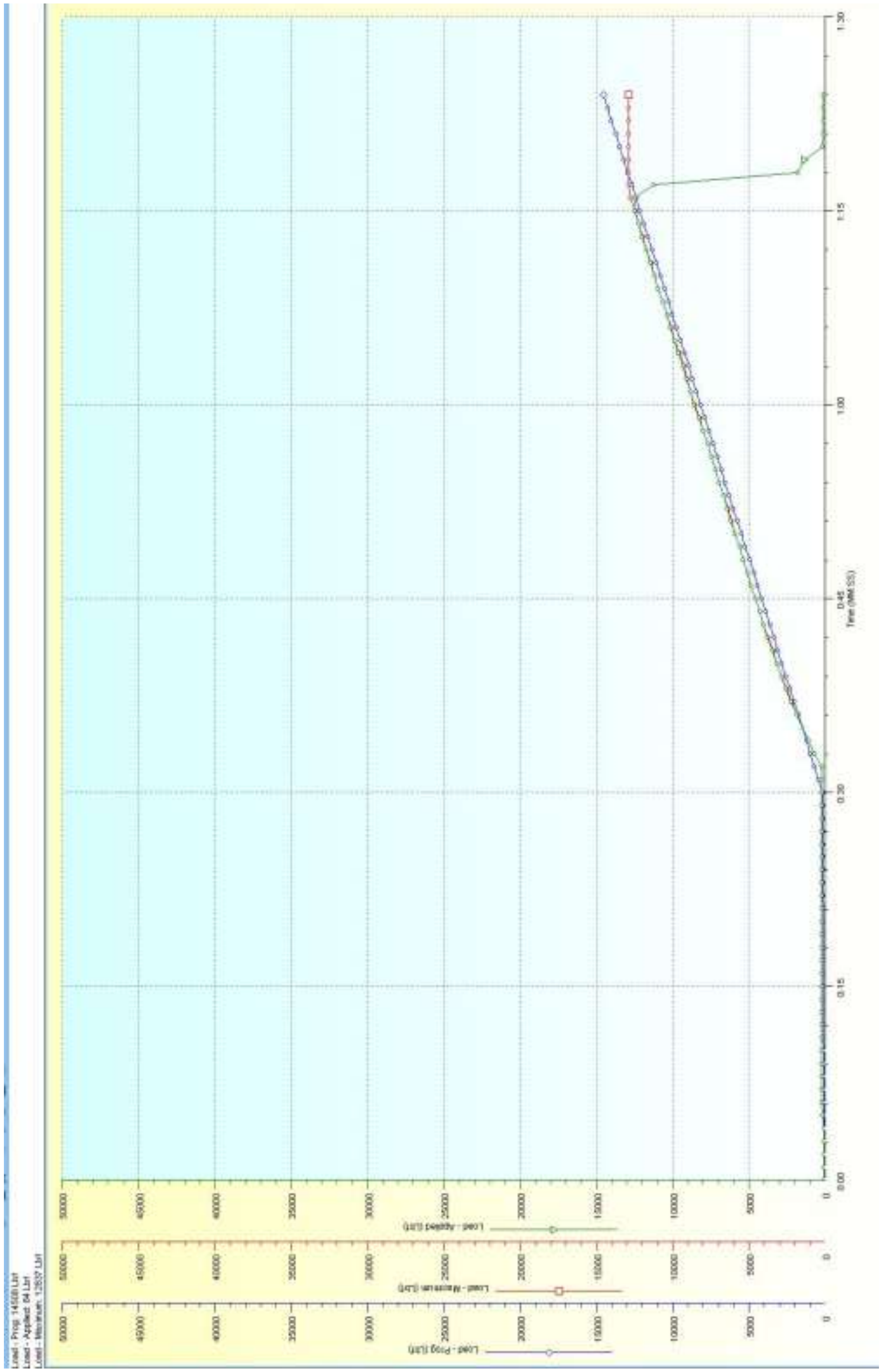


Figure A.8: Cement sample having 2.0 % Nano silica under compressive strength test



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